ARIZONA DEPARTMENT OF TRANSPORTATION

USE OF CHINLE CLAY AND MODERATE HEAT FOR THE PRODUCTION OF SYNTHETIC AGGREGATE

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16. Abstract

In 1972, the Arizona Department of Transportation (Highway Division) initiated a research project entitled "Use of Chinle Clay and Moderate Heats for the Production of Synthetic Aggregate". This project was sponsored by the Federal Highway Administration.

A testing program was laid out to determine what conditions and factors affected the strength of the synthetic aggregate produced from Chinle Clay.

The factors considered in the experimental program were: Temperature of sintering, Time of sintering, Moisture content, Clay content and wet density.

The tests showed that the main factors affecting the strength were temperature and wet density. More over, the results show that while it is not possible to use moderate heats, i.e., temperatures ranging from 204°C (400°F) to 650°C (1200°F), it is possible to produce a strong aggregate from Chinle Clay with a temperature of 1100°C (1900°F).

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Hubert Rosenstock

CHAPTER 1

INTRODUCTION

Sponsorship

This report is based on work performed by the Materials Services, Division of the Arizona Department of Transportation, Phoenix, Arizona under contract with the Federal Highway Administration to investigate "Use of Chinle Clay and Moderate Heats for Production of Synthetic Aggregates." Funds for the project were provided by the Arizona Department of Transportation, in cooperation with the Federal Highway Administration.

Nature of Problem

Aggregates comprise much of the material required for highway construction and maintenance. Base course and surface courses may contain 90 to 100 percent aggregate. Bituminous base, binder and surface courses may contain more than 90 percent aggregate. Portland cement concrete averages about 75 percent aggregate.

The rapid growth in both highway construction and maintenance has increased the demand for high quality aggregate materials. Although there are extensive aggregate supplies in the southern half of state, aggregate supplies are virtually nonexistent in the northern half of Arizona. In the areas of the north where aggregates are economically available, every effort should be made to conserve these naturally existing aggregates for future use in the most expedient way.

Northern Arizona is covered by deposits of expansive Montmorillonite clay. This formation is called the Chinle Clay and has approximately 6,000 square miles of surface exposure. Throughout this area the clay has many colors ranging from light gray to dark purple. Although one will find this material in a wide range of colors, the engineering properties are quite similar. While this material is not suitable for use as an aggregate in its naturally occurring form, it was hoped that it could be processed into a suitable aggregate by thermal processing using moderate temperature ranges, i.e. 400°F (204°C) to 1200°F (650°C).

Approach to the Problem

The basic approach to the problem was to produce in the laboratory sufficient aggregate for evaluation using the thermal processing methods of Chinle Clay. These methods would be different from the existing process of manufacture of light weight aggregate from clay in that lower temperatures would be employed. This study would determine the lowest temperature sufficient to produce an agglomerate that would have resistance to attack by water and have the required strength. The use of the lower temperatures would result in lower fuel consumptions, greater production through a given furnace or kiln and equipment that is simple to operate.

In addition to the laboratory work a suitable amount of aggregate was produced by the Texas Transportation Institute, a non-profit research organization. It should be pointed out here that the "moderate temperature" range of 200°C (390°F) to 650°C (1200°F) was not possible to use and produce satisfactory synthetic aggregate from Chinle Clay material; temperature range limits had to be nearly doubled in order to obtain satisfactory results.

Object of Research

The object of this research is to develop new manufacturing methods or modifications of existing methods for the production of aggregates from Montomorillonite clay or clay shales without requiring excessive temperatures for transformation.

Scope of Report

The report on the use of Chinle Clay and Moderate Heats for Production of Synthetic Aggregates is presented in nine chapters. These chapters explain in detail the various areas of laboratory work as well as commercial-like production of synthetic aggregates conducted since September 29, 1972. Chapter 2 discusses geology of the site and a general background as to the type and quantity of material encountered in the area. Chapter 3 discusses the Developmental Procedure and Sample Preparation of the aggregate. Chapter 4 discusses laboratory work and preliminary as well as final evaluation of the parameters affecting the production of aggregate. Chapter 5 is a general discussion of the Statistical Methods utilized in the data analysis.

Chapter 6 is a brief outline of work done on fly-ash/chinle clay synthetic aggregate.

Chapter 7 gives an outline of some sintering work done on chinle clay and "Winslow Blow Sand".

Chapter 8 deals with the commercial-scale production of the synthetic aggregate by the Texas Transportation Institute.

Chapter 9 is a discussion of the conslusions and recommendations for future work.

CHAPTER II

GEOLOGY OF THE AREA AND SITE

The site is located in Northeastern Arizona on the Colorado Plateau geological province (see plate 1 on page 74). The nearly horizontal sandstones and shales of Triassic and Jurassic age in this area are intermittently covered by thin-bedded Tertiary sandstones and shales. This region is characterized by low, broad mesas overlooking wide, flat, stream valleys containing Quaternary to Recent sands and silt alluvium with low terraces containing a very limited quantity of gravel.

U.S. Interstate 40, each of Winslow, Arizona, traverses primarily the Triassic Chinle Formation as it crosses this area. The Chinle Formation is a part of the Triassic rocks of the Colorado Plateau. The triassic rocks commonly contain varieties of illite, kaolinite, chlorite and mixed-layer combinations of illite and montmorillonite or of chlorite and montmorillonite; Palygorskite is present in a few samples.

The Chinle Formation occurs throughout the Colorado Plateau and is noted for its abundance of fossile wood which is conspicuously exposed in the Petrified Forest National Park and also for the "Painted Desert." The Chinle Formation is separated into several members on the basis of lithologic differences. These various member groups of the Cinle Formation characteristically do not have sharp deliniations but grade both laterally and vertically into each other.

The site chosen for this study was in the region lithologically characterised as the Petrified Forest and Owl Rock members of the Cinle Formation. The dominant lithology of the Petrified Forest member is vari-colored swelling clay-stone with a thickness ranging from 0-300 meters, while the dominant lithology of the

Owl Rock member is composed primarily of pale-red, purple, and brown calcareous siltstones and claystones. It thins southward from Utah and intertongues with the upper part of the Petrified Forest member with a maximum thickness of 100 meters at the test site.

The Petrified Forest member extends over the southern part of the Colorado Plateau, while the Owl Rock member is present throughout most of southeastern Utah and in adjacent parts of Monument Valley in Arizona.

These two members of the Chinle Formation Formation closely resemble each other with regard to the clayey-minerals contained in each; hence, the location of the test site can be said to lie primarily in the Petrified Forest member.

The greatest portion of the rocks comprising the Petrified Forest member contain large amounts of smectites (montmorillonites). The claystones commonly contain mixed-layer montmorillonites and the coarser grained rocks commonly contain even larger amounts of montmorillonites. Of course, there are many variations in the clayey composition of these rocks, that is, there are regions of chloritic rocks mainly in the eastern portion of the Petrified Forest member and palygorskite-bearing rocks in the upper part of the Petrified Forest member. Similar palygorskite-bearing rocks are found in the lower part of the Owl Rock member.

The term "Chinle Clay" is used throughout this report to designate a sample of fine grained soil obtained from the Chinle Formation that exhibits a high degree of plasticity and a significant volume increase on contact with water. It should be noted that the variation of smectite content of samples obtained from the region of the test site can range from ten percent by weight to forty percent by weight.

Moreover, while it has not been specifically measured for any group of chinle clay samples, the coefficient of variation of smectite content can be expected to be large. Other constituents in a soil sample from the test site region are significant amounts of sandy silt, minor amounts of illite and mica.

In general, physical and chemical characteristics of sedimentary deposits are dependent for the most part on three factors: (1) foremost, of course, the parent material, (2) method of transportation of sediments, and (3) changes after deposition. Several depositional environments are responsible for the characteristics of the Chinle Formation. It is held that the conditions under which the Chinle was deposited were wholly continental--probably those of a well-graded but rather arid plain across which streams meandered and on which there were perhaps scattered lakes. Conglomerates of resistant materials transported from a great distance are scarce; hence, it is evident that the stream gradients were low. Continental origin is evidenced by the presence of fresh-water fossils, land vertebrates and the trees of the Petrified Forest. Evidence is present for the existence of a warm marine environment in portions of the area in which the Chinle outcrops. Montmorillonoid and bentonitic clay minerals are suggestive of volcanic activity at the time of the warm marine climate. The alteration of volcanic ash in such an environment is favorable to the formation of the aforementioned clays.

The weathered material on the slopes of the cuts extends to a depth of about one foot. This material has the typical reticulated appearance of an expansive clay, weathered in place from the parent material. The underlying material is very hard and brittle. The outcrop is fractured, slickensided, and shows no visible bedding planes.

CHAPTER III

DEVELOPMENTAL PROCEDURE

General

The scope and purpose of the developmental work was to locate a site where sufficient quantities of Chinle clay were available. The purpose of this was the anticipation of commercial development of the site if the research merited the economical investment necessary for the production of synthetic aggregate.

Site Selection and Sampling

The field work commenced in October 1972, with a visual survey of the area on Interstate 40 east of Holbrook, Arizona in an attempt to locate a sufficiently large volume of Chinle clay. After investigating various locations it was decided to select an area approximately one mile west of the Keams Canyon Interchange and 2000 feet south of I-40. This area is estimated to contain well over 1,000,000 cubic yards of exposed Chinle clay.

In December, 1972 about 2 cubic yards of the material was obtained by the Arizona Highway District personnel. This material was shipped to the Materials Services in Phoenix where most laboratory tests were performed.

Sample Preparation

Starting with the original soil sample obtained from the site, the material was spread in a four-inch layer on a concrete surface and allowed to air dry, it was crushed in a ball mill located at the Materials Services of the Highway Division of the Arizona Department of Transportation. The material was broken down to pass a #40 sieve and this material was stored for future testing. At all times care was taken to insure that the total sample was well mixed and uniform before storage. This enabled a homogeneous sample to be obtained throughout the testing program.

Physical Testing

The first tests performed on the clay were the index properties and grain size distribution. The material is very close to the border between MH or CH material on the Unified Classification Chart and as a CL according to the AASHTO system. The sample has a LL = 57; PL = 31; PI = 26; and a specific gravity = 2. Ninety-nine percent of the sample passes the #4 sieve and 32 percent passes the 200 sieve as received from the field.

A density test was performed in accordance with Arizona Test Method No. 232. The results were maximum dry density 101 lbs/ft^{3*} at 19% optimum moisture.

*($1620\ kg/m^3$).

CHAPTER IV

LABORATORY WORK

General

The chemical portion of this study was initiated upon receipt and preparation of the 2-cubic-yard sample from Holbrook in December 1972. Tests were performed to determine the chemical composition of the *in situ* clay in terms of Wt - % oxide. Control samples were also used for comparision, they were U.S.P. Bentonite and Wyoming Bentonite.

Chemical Analysis

Each sample was analyzed on a Perkin-Elmer Model 306 atomic absorption spectrophotometer and its composition determined by *Analytical Methods for Atomic Absorption Spectrophotometry*, Perkin-Elmer part No. 303-0152. The results are reported in terms of Wt - % oxide and are given in tables 7 through 12 contained in Appendix A.

Sample Preparation

The following standard procedure was used for sample preparation.

All samples for chemical analysis were assigned a pre-drying cycle which consisted of drying to constant weight at 105±5°C (221°F ± 9°F).

Additional Wt - % Oxide Analysis

In tables 10 and 11, Wt - % oxide analysis of "cores" of synthetic aggregate samples are presented. The analysis of these cores was conducted because during compressive testing it was observed that upon compressing the specimen to destruction, a central portion, called the core, remained intact.

This central portion was usually darker in color than the outer and apparently weaker portions of the aggregate. The color usually varied from a pale blue to a dark grey color. Under microscopic examination, it appeared to be composed of dark "glassy" appearing bubbles.

The cores that were selected for analysis were very carefully separated from the outer portions of the test specimen.

Only the largest whole cores were selected for testing. The assumption being that this material represented the strongest portion of the aggregate; hence, a chemical analysis of the Material seemed in order.

Differential Thermal Analysis

Differential thermal analysis tests were performed by the Materials Services, Arizona Department of Transportation, on various materials. The resulting thermograms are shown in Figures 1 through 6 in Appendix A.

SINTERING PROCESSES AS APPLIED TO CHINLE CLAY SAMPLES

Since this project utilized the techniques of sintering as applied to the production of synthetic aggregate an overview of sintering technology is given here.

Sintering is, of course, one part of the overall technique of Powder Metallurgy which is the technology of forming metal and non-metal powders into finished or semi-finished products by mechanical and thermal operations. These operations are performed at temperatures below the melting or sublimation points of at least the major portion of the material composition.

The main operations of powder metallurgic processing are:

1. The compacting process, during which suitably prepared powder mixtures are subjected at normal ambient or elevated temperatures, to a considerable compressive force, forming "green compacts".

2. The sintering process, in which the pressed "green compacts" are subjected to a thermal treatment which impacts to them the required mechanical strength as well as other desired properties.

SINTERING TECHNOLOGY AS APPLIED TO PRODUCTION OF SYNTHETIC AGGREGATE

Compacting the clay into "green compacts" of various sizes was to be accomplished by pouring the clay material into dies and subjecting it to the action of one or more plungers operated by a hydraulic or mechanical press.

Since the "green compacts" that would be formed for fabrication of synthetic aggregate would all relatively thin and of uniform height, the clay material may be compacted by "single action". That is by compression from one side only.

In this case the press operation, die and plunger design are very simple, consisting of a tubular die closed at one end and a simple piston type plunger, manually operated.

Moreover, in the very special case of clays, the sintering can be performed by extruding the clay material with a carbon or alcohol binder at high pressures through appropriate dies; and subsequently heating the extruded material to volatilize the carbon or alcohol binder and sinter the powder. The carbon or alcohol binder serves as an additional energy source during the sintering process.

After leaving the press, the "green compacts" are then transferred to a sintering furnace. These were the processes utilized throughout this project for the production of the lab specimens of synthetic aggregate and, indeed, the process applied by T.T.I. to produce the synthetic aggregate from chinle clay.

The main purpose of the sintering process is the transformation of the "green compact", consisting of individual particles, to a mass in which the particles have lost their identity and recrystallization and grain growth have taken place across the former particle boundaries. Since the chinle clay is a hetrogeneous mass, another function of the sintering process is material homogenization by diffusion.

Conditions which can be controlled during the sintering operations are:

1. The compacting operation.

2. The rate of heating

3. The maximum sintering temperature

4. The length of the actual time of sintering ("soaking period") - length of time which the "green compact" is kept at maximum temperature.

5. The rate of cooling (this is a vital parameter which, due to a lack of sufficient laboratory time, was not explored thoroughly.

The optimum conditions for the production of specific mechanical properties and dimensions of the sintered part depend, however, not only on the sintering conditions but also, to a large extent, on the properties of the "green compact", which in turn are determined by the powder characteristics and conditions under which the material is compacted.

This was in fact borne out vividly by our experience both in the lab and with the T.T.I. product.

From our lab studies and the results of test performed on the T.T.I. material, the following factors were seen to have the greatest influence in determining the final result of a powder metallurgic production of synthetic aggregate. They are given as follows:

1. The physicochemical characteristics of the clayey soil used as a raw material; i.e.:

a. Susceptibility of the chinle material to plastic deformation, which is a direct function of its physicochemical characteristics.

b. Size distribution, shape and porosity of the clayey material together with other characteristics, such as apparent density, compression ratio and flow, which are mainly determined by particle size, shape and porosity.

c. The actual % by Wt clay content of the "chinle clay" samples and the nature as well as distribution of the impurities. The natural clay contents of samples varied greatly, from 10% by Wt to 40% by Wt., with an average of 24%.

2. The compacting conditions, i.e.:

a. Maximum pressure applied upon the clay material during the compacting.

b. Timing of the pressure application - that is to say, the rate of pressure increase during the compression and the time during which the maximum pressure is maintained.

c. Shape of the compact or die cavity.

d. Manner in which the pressure is applied upon the clay material.

e. Temperature at which the compression is performed.

f. Moisture content* of the clay and lubrication of the die walls.

g. Die material.

3. The sintering conditions:

a. Maximum temperature.

b. Timing of heat treatment, characterized by the time-temperature curve of the entire sintering cycle (rate of heating, time during which the maximum temperature is maintained, rate of cooling).

c. Sintering atmosphere.

d. Pressure applied to the compact during sintering.

e. Type of heat supply.

* This could also be listed under Item 1.

It is to be stressed that the most important effect of sintering conditions for the case of the hetrogeneous chinle clay is homogenization by diffusion. Diffusion is also involved in the sintering of homogeneous powders, particularly in recrystallization and grain growth. However, in the case of heterogeneous powders, consisting of more than one component, the effect of diffusion becomes preponderant, which in the case for chinle clay materials.

Many theoreticians have and are working on models of the mechanism of sintering. The following preliminary conclusions are based on complete laboratory studies. It should be stressed that these conditions are not to be constructed as general physical laws but guidelines.

1. At temperatures below $A = 0.23$ (where A designates the fraction of the absolute melting or sublimation temperature)* the main effect of heat treatment is a general increase in adhesion and surface stability. Surface stabilization results in a reduction of the surface area available for absorption of gases such as N_2, Ar, etc.

2. At temperatures between $A = 0.23$ and $A = 0.36$, surface diffusion causes a general increase in surface activity. The chemical potential increases, and the surface area available for absorption as well as the volume of capillaries is enlarged. In spite of the increase of accessible surface, a general loosening of the surface structure may result in the release of previously absorbed gases.

3. Between $A = 0.33$ and $A = 0.45$, activation by surface diffusion is practically completed and continued surface diffusion and increasing adhesion produce stabilization (deactivation). Above $A = 0.41$ shrinkage may take place, although for the chinle clay samples tested an increase in volume was at times noted.

* Our experiments did not allow us to obtain a definite figure for the clay samples used, but the figure may be around 1400^o c - 2000^o c.

4. Between A = 0.37 and A = 0.53, the surface atoms are rearranged by internal lattice diffusion and reactivation becomes apparent in marked increases of chemical potential, surface area and volume of accessible capillaries (i.e., the material "blossoms" out). At A = 0.52 last traces of volatile constituents are expelled.

5. Between A = 0.48 and A = 0.80 continued lattice diffusion, tending toward formation of single crystals, causes deactivation.

6. Above A = 0.80 reactivation is observed and interpreted as a stage preliminary to the melting sublimation process.

These preliminary conclusions are as fas as our necessarily limited research has taken us, regarding the applications of the sintering process to the production of synthetic aggregate.

It is strongly felt that more effort along these lines would provide a viable and economical method for the production of lightweight synthetic aggregate from Chinle Clay material.

PRELIMINARY SCINTERING

Dr. Charles O'Bannon's previous work with soil samples from this test site indicated, via hydrometer analysis, that the average clay content was about 24% by wt., with many samples having as much as 40 percent clay content.

Moreover preliminary samples composed of samples of 24% clay and samples enriched by additions of sufficient amounts of Wyoming Bentonite to bring the clay content up to 40% by wt. were sintered at 200°C (390°F) and 600°C (1120°F). It was found that these samples, so treated, exhibited little or no increase in strength.

Based on this preliminary work the levels of the variables that effect the production of synthetic aggregate were determined. It was decided that for the first burn to use temperatures of 900°C and 1100°C, scintering time was 15 and 35 minutes, with a clay content of 24 and 40%, density of 90 and 110 pounds per cubic foot and moisture content of 20 and 30%. For the second burn all levels were the same except the levels of temperature. For the second burn the range of temperature was 600°C and 700°C. The clay content of 40% was artifically induced by addition of sufficient amounts of U.S.P. Wyoming Bentonite material.

With the levels of variables established the tests were completed in the following manner. A known weight of dry precrushed Chinle Clay was mixed with the required amount of water to bring it up to the desired moisture content. The material was thoroughly mixed and then statically compacted to a known volume to obtain the required density. The samples were initially 1.25 inches in diameter by 2.5 inches high*;however, too much dislocation occurred during firing, therefore, the height of the sample was reduced to 1.25 inches. When this modification was incorporated into the design the burn was completed without further difficulties. In this manner 96 samples were compacted, fired and their compressive strength determined. The process was repeated for the second burn and all data was analyzed as indicated in the statistical chapter which is in Chapter 5 of this report.

*(3.17 cm diameter X 6.35 cm high)

CHAPTER V

SYNOPSIS OF STATISTICAL DESIGNS AND METHODOLOGY USED

One of the experimental objectives of this proj-ct was to determine the significant factors influencing the aggregate strength from the set of variables:

T = firing temperature

t = time of firing at temperature T

C_c = clay content

D = wet density

M_c = moisture content

And to quantitatively express the influence of these factors on the aggregate strength with a functional relation

$$S = f(M_c, C_c, D, T, t)$$

Letting

$$X_1 = T, X_2 = t_c, X_3 = C_c, X_4 = D \;\&\; X_5 = M_c$$

then

$$S = f(y_1, y_2, y_3, y_4, y_5)$$

Now it is also assumed that $S = f(y_1, y_2, y_3, y_4, y_5)$ is a well-behaved function so that it may be expanded into a Taylor's series at any given point ($y_1 = y_1^1$, $y_2 = y_2^1$, $y_3 = y_3^1$, $y_4 = y_4^1$, $y_5 = y_5^1$). So that to the second order where $y_1^1 = y_1 + \xi_1$

$$f(y_1 + \xi_1, y_2 + \xi_1, y_3 + \xi_3, y_4 + \xi_4, y_5 + \xi_5) = f(y_1, y_2, y_3, y_4, y_5) +$$

$$\sum_{i=1}^{5} \partial_i f(y_1, y_2, y_3, y_4, y_5)\xi_i + \tfrac{1}{2} \sum_{i,j=1}^{5} \partial_{ij} f(y_1, y_2, y_3, y_4, y_5)\xi_i \xi_j + \cdots$$

Thus we may approximate S to varying degrees of accuracy with a polynomial, as is well known. Hence the experimental data was collected and an attempt was made to "fit" a first or second degree polynomial to the data.

It was decided to run the experiment as a "Factorial Experiment".

Now a factorial experiment is one in which all levels of a given factor are combined with all levels of every other factor in the experiment. The advantages of a factorial experiment are as follows:

(1) More efficiency than one-factor-at-a-time experiments.

(2) All data are used in computing the effects of the factors on the material strength.

(3) Information is gleaned on possible interaction between factors. For example there may be a great deal of interaction between the wet density and temperature of the burn on the final material strength.

Clearly as the levels of the factors, T, t, C_c, D and M_c are increased these advantages become more pronounced.

In order to facilitate the analysis of experimental data it was decided to initially conduct the experiments as 2^5 factorial design matrix written in Yates Order, see Figure 7 . Although this may seem like a trivial case, since only two levels are involved, it is useful to illustrate what main effects and interaction are really involved with the smallest amount of computational time used.

The five factors T, t, C_c, D and M_c are identified as x_1, x_2, x_3, x_4, x_5 respectively and their high and low levels as plus and minus signs, + and −, respectively.

In the 2^K factorial design one has the following "factorial" effects:

<u>5 MAIN EFFECTS</u>

5(5−1)/2=10	two factor interaction effects
5(5−1)(5−2)/2.3=10	three factor interaction effects
5(5−1)(5−2) (5−3)/2.3.4=5	four factor interaction effects
5(5−1)(5−2) (5−3) (5−4)/5=1	five factor interaction effects

These, together, yield a total of 31 factorial effects for the case of the 2^5 factorial.

The analysis of the experimental data was performed by considering the effects of the various design parameters on the response by calculating their <u>contrasts</u>.

The contrast of the factors is a direct measure of their effect on the response of the dependent variable, here the strength of the material.

The statistics known by the term contrasts are defined as follows:

Given i, j, . . ., k factors each at two levels with r replications of the experiment then the <u>contrast</u> of the i, j, . . ., k effect is given by:

$$(\text{CONTRAST}) = \frac{1}{r2^{k-1}} (i, j, \ldots, k) \cdot T$$

where (i, j, . . ., k), called the <u>contrast coefficient</u> matrix, stands for the 1×2^k matrix of plus and minus signs displayed in the appropriate column in Table 1, i.e. if we are estimating the 4-way interaction of the variables i, j, . . ., k = 1234 then (1234) = (+ - - - - + + + + - + + - + - - - + - + - + + + + - - + - - - +).

T stands for the $2^K \times 1$ total treatment matrix.

That is each entry of T is a grand total of the replications of the experiment with, say the variables 1, 2, 3, 4 and 5 set at their lowest values, then all but 4 at their lowest, etc. until all 32 elements of T are obtained. Note that T is the same for the calculation of any contrast, i.e. for the main effects to the 5-way interaction term, in addition, r denotes the number of times the experiment was replicated.

The table of contrast coefficients, called Table 1, is prepared as follows:

One first notes that Figure 7 displays the 5 variables or parameters where the minus signs correspond to the lowest value assigned to that variable and the plus sign the highest. Then for example to obtain, say, the entry of Table 1 in row three of the contrast coeeficient column labled (12), we have that the sign entry in column (1) row three is (-) and the sign entry in column (2) row three is (+), and hence the entry for row three in column (12) is (+) (-) = -. All other entries for the various interactions are obtained similarly. The completed contrast coefficeints for all of the possible interactions are displayed in Table 1. A sample calculation for determing the estimated for the interaction is presented in Tables 2A and B.

THE DESIGN MATRIX FOR THE 2^5 FACTORIAL
IN
YATE'S ORDER

RUN #	X_1	X_2	X_3	X_4	X_5
1	−	−	−	−	−
2	+	−	−	−	−
3	−	+	−	−	−
4	−	−	+	−	−
5	−	−	−	+	−
6	−	−	−	−	−
7	+	+	−	−	−
8	+	−	+	−	−
9	+	−	−	+	−
10	+	−	−	−	+
11	−	+	+	−	−
12	−	+	−	+	−
13	−	+	−	−	+
14	−	−	+	+	−
15	−	−	+	−	+
16	−	−	−	+	+
17	+	+	+	−	−
18	+	+	−	−	+
19	+	+	−	+	−
20	+	−	−	+	+
21	+	−	+	+	−
22	+	−	+	−	+
23	−	+	+	−	+
24	−	−	+	+	+
25	−	+	−	+	+
26	−	+	+	+	−
27	+	+	−	+	+
28	+	+	+	+	−
29	+	−	+	+	+
30	+	+	+	−	+
31	−	+	+	+	+
32	+	+	+	+	+

FIGURE 7

CONTRAST COEEFICIENTS FOR 2^5 FACTORIAL DESIGN

MAIN EFFECTS					2-WAY INTERACTION														3-WAY INTERACTION							4-WAY INTERACTION				5-WAY
					1				2			3		4																
1	2	3	4	5	2	3	4	5	3	4	5	4	5	5	123	125	135	134	145	124	235	345	245	234	1245	1234	1345	1235	2345	12345
−	−	−	−	−	+	+	+	+	+	+	+	+	+	+	−	−	−	−	−	−	−	−	−	−	+	+	+	+	+	−
+	−	−	−	−	−	−	−	−	+	+	+	+	+	+	+	+	+	+	+	+	−	−	−	−	−	−	−	−	+	+
−	+	−	−	−	−	+	+	+	−	−	−	+	+	+	+	+	−	−	−	+	+	−	+	+	−	−	+	−	−	+
−	−	+	−	−	+	−	+	+	−	+	+	−	−	+	+	−	+	+	−	−	+	+	−	+	+	−	−	−	−	+
−	−	−	+	−	+	+	−	+	+	−	+	−	+	−	−	−	−	+	+	+	−	+	+	+	−	−	−	+	−	+
−	−	−	−	+	+	+	+	−	+	+	−	+	−	−	−	−	+	−	+	−	+	+	+	−	−	+	−	−	−	+
+	+	−	−	−	+	−	−	−	−	−	−	+	+	+	−	−	+	+	+	−	+	−	+	+	+	+	−	+	−	−
+	−	+	−	−	−	+	−	−	−	+	+	−	−	+	−	+	−	−	+	+	+	+	−	+	−	+	+	+	−	−
+	−	−	+	−	−	−	+	−	+	−	+	−	+	−	+	+	+	−	−	+	−	+	+	+	+	+	+	−	−	−
+	−	−	−	+	−	−	−	+	+	+	−	+	−	+	+	−	−	+	−	+	+	+	+	−	+	−	+	+	−	−
−	+	+	−	−	−	−	+	+	+	−	−	−	−	+	−	+	+	+	−	+	−	+	+	−	−	+	−	+	+	−
−	+	−	+	−	−	+	−	+	−	+	−	−	+	−	+	+	−	+	+	−	+	+	−	−	+	+	−	−	+	−
−	+	−	−	+	−	+	+	−	−	−	+	+	−	−	+	−	+	−	+	−	−	+	−	+	+	−	−	+	+	−
−	−	+	+	−	+	−	−	+	−	−	+	+	−	−	+	−	+	−	+	+	+	−	+	−	−	+	+	−	+	−
−	−	+	−	+	+	−	+	−	−	+	−	−	+	−	+	+	−	+	+	−	−	−	+	+	−	−	+	+	+	−
−	−	−	+	+	+	+	−	−	+	−	−	−	−	+	−	+	+	+	−	+	+	−	−	+	+	−	+	−	+	−
+	+	+	−	−	+	+	−	−	+	−	−	−	−	+	+	−	−	−	+	−	−	+	+	−	+	−	+	−	+	−
+	+	−	−	+	+	−	−	+	−	−	+	+	−	−	−	+	−	+	−	−	−	+	−	+	−	+	+	−	+	+
+	+	−	+	−	+	−	+	−	−	+	−	−	+	−	−	−	+	−	−	+	+	+	−	−	−	−	+	+	+	+
+	−	−	+	+	−	−	+	+	+	−	−	−	−	+	+	−	−	−	+	−	+	−	−	+	−	+	−	+	+	+
+	−	+	+	−	−	+	+	−	−	−	+	+	−	−	−	+	−	+	−	−	+	−	+	+	−	−	−	+	+	+
+	−	+	−	+	−	+	−	+	−	+	−	−	+	−	−	−	+	−	−	+	−	−	+	+	+	+	−	−	+	+
−	+	+	−	+	−	−	+	−	+	−	+	−	+	−	−	−	−	−	+	+	+	+	−	−	−	+	+	+	−	+
−	−	+	+	+	+	−	−	−	−	−	−	+	+	+	+	+	−	−	−	+	−	+	−	−	+	+	−	+	−	+
−	+	−	+	+	−	+	−	−	−	+	+	−	−	+	+	−	+	+	−	−	−	−	+	−	−	+	+	+	−	+
−	+	+	+	−	−	−	−	+	+	+	−	+	−	−	−	+	+	−	+	−	−	−	−	+	+	−	+	+	−	+
+	+	−	+	+	+	−	+	+	−	+	+	−	−	+	−	+	−	−	+	+	−	−	+	−	+	−	−	−	−	−
+	+	+	+	−	+	+	+	−	+	+	−	+	−	−	+	−	−	+	−	+	−	−	−	+	−	+	−	−	−	−
+	−	+	+	+	−	+	+	+	−	−	−	+	+	−	−	+	+	+	+	−	−	+	−	−	−	−	+	−	−	−
+	+	+	−	+	+	+	−	+	+	−	+	−	+	+	+	+	+	−	−	−	+	−	−	−	−	−	−	+	−	−
−	+	+	+	+	−	−	−	−	+	+	+	+	+	+	−	−	−	−	−	−	+	+	+	+	−	−	−	−	+	−
+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+

TABLE 1

TABLE 2-A

SAMPLE CALCULATION OF CONTRAST

In each experimental run, #'s 1, . . ., 32, there are three replications. Thus in the formula for calculating the contrast.

$$\text{(CONTRAST)} = C = \frac{1}{r2^{k-1}} \; (i, j, \ldots, k) \cdot T = \frac{1}{3 \times 2^4} \; (i, j, \ldots, k) \cdot T$$

Now T represents the treatment total 32 x 1 matrix , see Table 2-B, r is the number of replications (in this case r = 3)

Now let's calculate the contrast for the 5-Way interaction term in the experimental statistical burn #1.

To effect this computation via the above formula, we go to Table 1 and look at the column corresponding to the 5-Way interaction 1 2 3 4 5. Now using a matrix notation we have the following 1 x 32 matrix.

(−,+,+,+,+,+,−,−,−,−,−,−,−,−,−,−,+,+,+,+,+,+,+,+,+,+,−,−,−,−,−,+)

and for T we have a 32 x 1 matrix

$$T = \begin{vmatrix} 7189 \\ 7623 \\ \cdot \\ \cdot \\ \cdot \\ 14091 \\ 12196 \\ 7405 \\ 10100 \\ 13737 \\ 10018 \end{vmatrix}$$

, ordinary matrix mul. and division by 48 then gives the contrast for the 5-Way interaction.

All of the other contrast are claculated in a similar manner from information contained in Table 1 and Table 2-B.

TABLE 2-B

TREATMENT TOTAL 32 x 1 MATRICES

FACTORS SET AT LEVELS CORRESPONDING TO		TREATMENT TOTAL 32 x 1 MATRICES STAT. BURN #1	STAT. BURN #2
Run	#1	7189	4064
	#2	7623	6262
	#3	10721	5165
	#4	9327	4055
	#5	20873	1216
	#6	19076	11595
	#7	8552	5949
	#8	8548	16749
	#9	13736	16466
	#10	12147	12898
	#11	11213	4098
	#12	18904	10581
	#13	17477	12763
	#14	22187	12898
	#15	13573	6909
	#16	15802	9876
	#17	8495	7618
	#18	11592	17592
	#19	14160	16029
	#20	14508	12654
	#21	12887	18600
	#22	8082	8198
	#23	13700	8223
	#24	17048	6961
	#25	17530	12031
	#26	23291	13852
	#27	14091	13020
	#28	12196	21509
	#29	7405	6386
	#30	10100	10604
	#31	13737	6832
	#32	10018	15500

After these computations were performed the next step in the analysis was to determine how many of the $(2^k - 1)$ factorial effects were in fact significant in determining the experimental response.

As is well known in many cases where k factors are being considered, it becomes clear after the experiments have been performed that h, where $h<k$, of the factors have very small or no effects on the response when compared with the remaining $(k - h)$ factors. When this occurs one can then go to a 2^{k-h} factorial replicated 2^h times thus providing $(2^h - 1)\ 2^{k-h}$ degrees of freedom for estimating σ_2.

Now in order to check with variables are the "real" main effects and which variables cause "effects" solely because of random fluctuations the following well established procuedure was utilized.

If the effects are due to random fluctuations, present during the experimental work, it follows that these fluctuations may be crudely looked upon as being normally distributed. This is what was done with our data, the responses were checked against the variations of the factors to see what factors contributed "random" effects only.

Now relatively simple way to check experimental data for membership in a normal population is as follows:

Let $x_1, x_2, \ldots, x_n$ denote n observed results, taken in the order in which the observations have been made and let $x_{(1)}, x_{(2)}, \ldots, x_{(n)}$ denote the same n results ranked in order of increasing magnitude. The frequency H(x) of observations that are smaller than or equal to x; $(i = 1, 2, \ldots, n)$ called the cumulative frequency, is

$$H(x) = \begin{cases} 0 \text{ for } x < x_1 \\ \frac{i}{n} \text{ for } x_{(i)} \leq x < x_{(i+1)} & : i = 1, 2, \ldots, n-1 \\ 1 \text{ for } x \geq x_n \end{cases}$$

H(x) is represented as a step curve, called the cumulative frequency polygon which increased from 0 to 1 in "jumps" of $\frac{1}{n}$ for $x_{(1)}, x_{(2)}, \ldots, x_{(n)}$. If several observations take on the same value, the "jump" is a multiple of $\frac{1}{n}$.

It should be noted that $H_{(x)}$ is <u>observed</u> value of the cumulative probability P(x). Moreover, H(x) deviates "at random" from P(x) and because of this it may be shown that the extreme points are less important than the midpoints. For example, we note the following, if $y = \Phi(x)$ is approximately linear then $y = \Phi(x) \approx \Phi(\xi) + (x - \xi)\,\phi'(\xi)$ now it is well known that if $y = A + Bx$ then the variation of y, i.e. $V(A+Bx) = B^2 V(x)$. Thus if $y \approx \phi(\xi) + (x - \xi)\,\phi'(\xi)$ we have $V(y) = (\phi'(\xi))^2 V(x - \xi)$. Thus we have, since we are assuming $H \approx P(\xi) + (u - \xi)\frac{dp^*}{du}$, where $H = i/n$ and $p = \phi(u)$ (error function) that: $V(H) \approx V\left\{(u - \xi)\right\}\left(\frac{dp}{du}\right)^2$, let $u - \xi = u_H$ then $V(H)/\left(\frac{dp}{du}\right)^2 \approx V(u_H)$, where $dp/du = \phi'(\xi)$.

Moreover, it is true that if we take the frequency $H = \frac{i}{n}$ as a random variable then

$$V\left(\frac{i}{n}\right) = P \times \frac{(1-P)}{n}$$

$$\phi(Up) = P$$

$$\phi(-UP) = (1-P)$$

i.e., the error function evaluated at the "fractile" Up.

Thus, since H is the frequency,

$$V(u_H) = \frac{P(1-P)}{n} = \frac{\phi(Up)}{n} \times \phi(-Up)$$

$$V(u_H) \approx \phi\,\frac{(Up) \times \phi(-Up) \times (\phi'(Up))^{-2}}{n}$$

*i.e., we are assuming that H(X) is approximately equal to the first two terms of the Taylor's series for the error function.

i.e., the variance of the fractile u_p corresponding to the cumulative frequency H(x) is inversely proportional to the number of observations. Also, the quantity depends only on u_p and is a minimum at $u_p = 0$ where p = 50%. nV (u_H) is symmetrical about $u_p = 0$ and increases as the numerical value of p - 0.5 increases. Thus one may loosely but correctly infer that the extreme points are less important than the midpoints when determining whether or not the data is approximately normally distributed.

Now in pratical work the cumulative step-curve is not drawn in full, but is indicated by n of its points and at the point $x = x(i)$, H(x) jumps from $\frac{i-1}{n}$ to $\frac{i}{n}$.

An obvious substitution for the step curve is therefore given by the n points $(x_{(i)}, \frac{i-1/2}{n})$ which are, of course, situated at the midpoints of the vertical parts of the step-curve. These, then are the points which are plotted on probability paper; i.e., the points $(x_{(i)}, \frac{u(i-1/2)}{n})$ are plotted in an (x, u) coordinate system. It has been shown by J. Ipsen and N. K. Jerne that while this method gives a systematic error this error is insignificant for n>25.

Following this procedure the $(2^5 - 1)$ estimates of the factorial effects were first ordered as $e_{(1)}, \ldots, e_{(2^k - 1)}$. Then these were plotted against

$$P(i) = \frac{(i-0.5)}{2^k - 1}$$

i = 1, . . ., 2 - 1 on normal probability paper. Now if the effects $e_{(i)}$ are due solely to "random" errors their graph is approximately a straight line.

However, the estimates of largest value will, if they reflect <u>real effects</u>, lie off the straight line. With this handy method one easily notes which factors in the experiment are significant.

The $e_{(i)}$ versus $P_i = (i-0.5)/31$ ordering for this experiment is shown in Tables 3A and 3B.

Now in order to simplify the chemico-engineering analysis it was decided at the onset of the testing that for a first approximation only, the significant main effects would be utilized as variables for subsequent testing. This, of course, a-priori ruled out further consideration of any interaction terms which would be statistically significant but not dominately so; i.e., did not lie significantly farther from the straight line than any main effects such as temperature or density- clearly if the graph indicated that purely interaction effects such as temperature-moisture, density-moisture, clay-moisture, clay-density-temperature, etc., were in fact the dominating effects our first stance would be untenable and hence would have to be abandoned. However, as the plotted data shows the interaction effects were not "greater" than two of the main effects due to temperature and density. All of the other main effects due to time, clay content and moisture content were domonstrably due to random fluctuation.

TABLE 3-A

RANKED CONTRAST

i	TREATMENT	CONTRAST	$\frac{(i-0.5)}{31}$
31	x_4	1478	0.984
30	x_{145}	642	0.952
29	x_{12}	630	0.919
28	x_2	537	0.887
27	x_{12345}	251	0.854
26	x_{245}	168	0.822
25	x_5	125	0.790
24	x_{123}	121	0.758
23	x_{135}	112	0.725
22	x_{235}	63	0.694
21	x_{1234}	49	0.661
20	x_{345}	36	0.629
19	x_{34}	11	0.596
18	x_{124}	-31	0.564
17	x_{234}	-32	0.532
16	x_{15}	-51	0.500
15	x_{1245}	-90	0.468
14	x_{25}	-95	0.435
13	x_{1235}	-103	0.403
12	x_{1345}	-117	0.371
11	x_{2345}	-226	0.339
10	x_{125}	-264	0.306
9	x_{13}	-316	0.274
8	x_{14}	-328	0.242
7	x_{23}	-389	0.210
6	x_{134}	-398	0.177
5	x_3	-458	0.145
4	x_{24}	-558	0.113
3	x_{35}	-728	0.086
2	x_{45}	-1031	0.048
1	x_1	-1226	0.016

Table 3-A

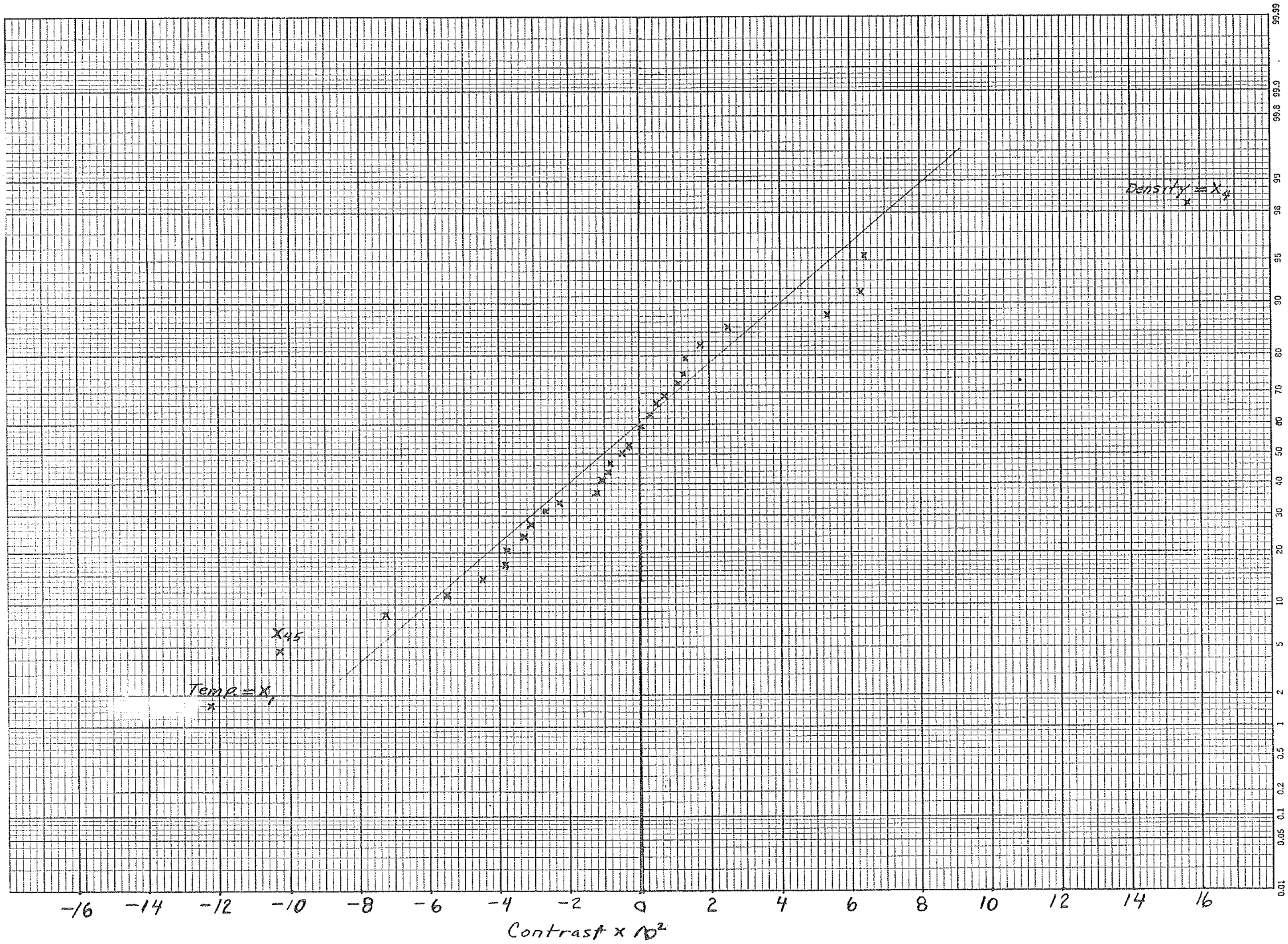
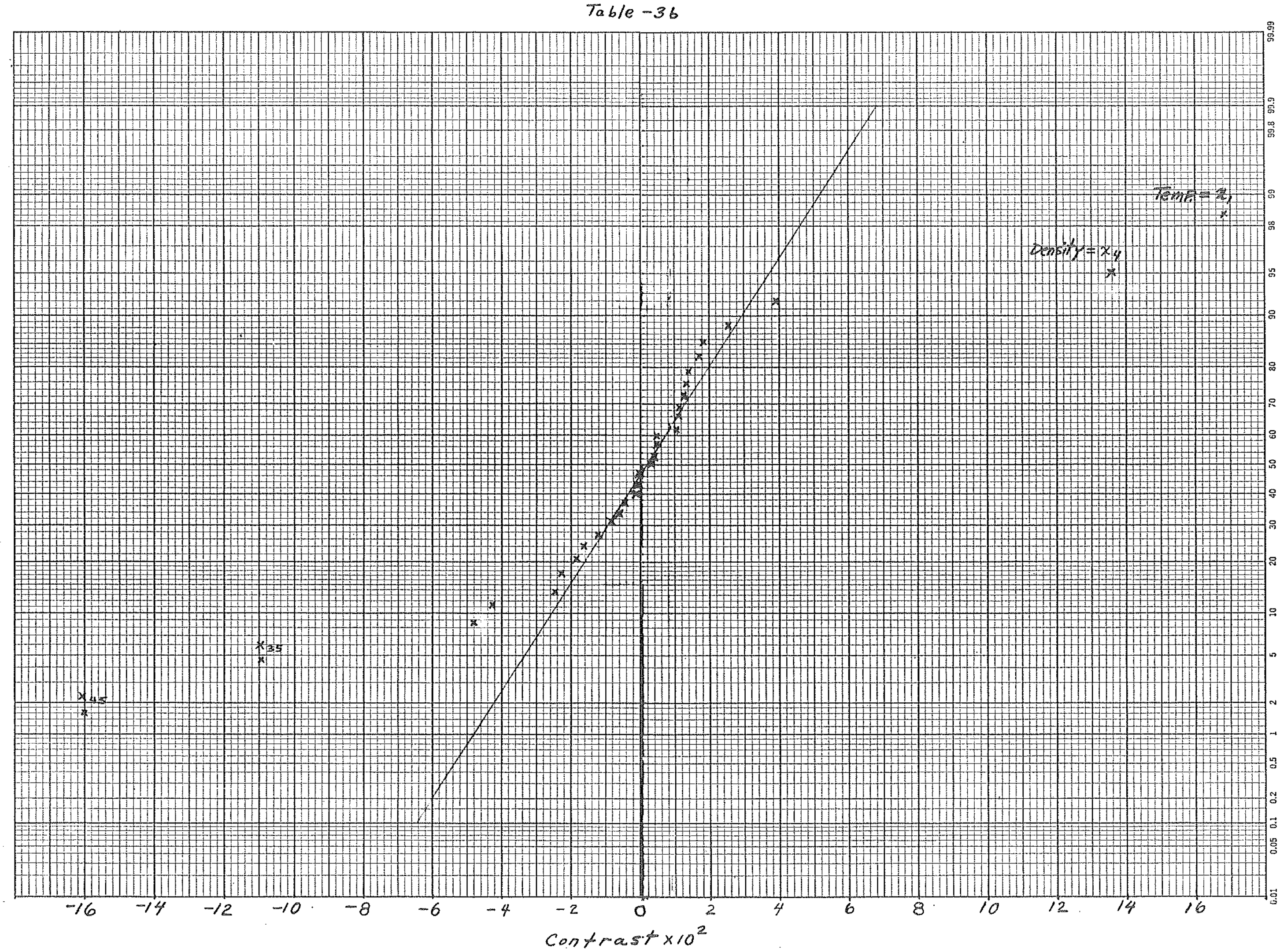

Density = X_4
X_{45}
Temp. = X_1
-16
-14
-12
-10
-8
-6
-4
-2
0
2
4
6
8
10
12
14
16
Contrast × 10^2
99.99
99.9
99.8
99
98
95
90
80
70
60
50
40
30
20
10
5
2
1
0.5
0.2
0.1
0.05
0.01

TABLE 3-B

RANKED CONTRAST

i	TREATMENT	CONTRAST	$\frac{(i-0.5)}{31}$
31	x_1	1640	0.984
30	x_4	1362	0.952
29	x_{34}	393	0.919
28	x_2	250	0.887
27	x_{14}	182	0.854
26	x_{124}	176	0.822
25	x_{123}	140	0.790
24	x_{134}	135	0.758
23	x_{23}	121	0.725
22	x_{1345}	112	0.694
21	x_5	110	0.661
20	x_{25}	103	0.629
19	x_{12345}	47	0.596
18	x_{2345}	44	0.564
17	x_{12}	40	0.532
16	x_{234}	37	0.500
15	x_{13}	-1	0.468
14	x_{1235}	-10	0.435
13	x_{1234}	-12	0.403
12	x_{345}	-54	0.371
11	x_{245}	-66	0.339
10	x_{24}	-83	0.306
9	x_{1245}	-121	0.274
8	x_{235}	-161	0.242
7	x_{125}	-184	0.210
6	x_{15}	-236	0.177
5	x_{135}	-255	0.145
4	x_{145}	-429	0.113
3	x_3	-489	0.086
2	x_{35}	-1099	0.048
1	x_{45}	-1601	0.016

Table -3b



Temp. = x_1
Density = x_4
35
45
-16 -14 -12 -10 -8 -6 -4 -2 0 2 4 6 8 10 12 14 16
Contrast $\times 10^2$
99.99
99.9
99.8
99
98
95
90
80
70
60
50
40
30
20
10
5
2
1
0.5
0.2
0.1
0.05
0.01

Thus to facilitate further testing it was decided that the factors to be used as main effects would be temperature and density.

With this result, a new experimental design was constructed. This was to determine a response equation based on the variables temperature and density.

Now it is well known that in order to fit the second order model in k variables a total of $\binom{k+2}{2}$ coefficients, a minimum of three levels of each of the variables x must be used. This, of course, suggested 3^k factorial design.

The design chosen for our purposes is displayed in Figures 8 and 9. The temperatures chosen in this case were 700^oC, 900^oC and 1100^oC and the wet densities were 115 lbs./cu. ft., 120 labs./cu/ ft/, and 125 lbs./cu. ft.

The experimental data yielded as surface as shown in Figure 10. Our corresponding equation $S = 491\ x_1^2 - 1542\ x_2^2 - 111\ x_1\ x_2 + 1210\ x_1 - 76\ x_2 + 5543$ fitted to the data yielded the surface shown in Figure 11. This result justified our use of the variables, temperature and density, as the significant main effects.

It should be stressed here that any such response equation fitted to experimental data is not a unique equation by any means. That is to say be changing the bounds on the region over which the temperature, density, etc., vary would result in a considerably different equation for the response surface.

However, within the region that our equation was fitted it was found to provide about ±10% predictive accuracy.

The critical points* for the equation were found to be $T = 886^oC$ and $D = 126$ lbs./cu. ft. Based on this information procedural instruction were given to Dr. Ledbetter at T.T.I. so that a preliminary batch of synthetic aggregate could

THE 3^2 FACTORIAL

K = 2 VARIABLES

X_1	X_2
–	–
0	–
+	–
–	0
0	0
+	0
–	+
0	+
+	+

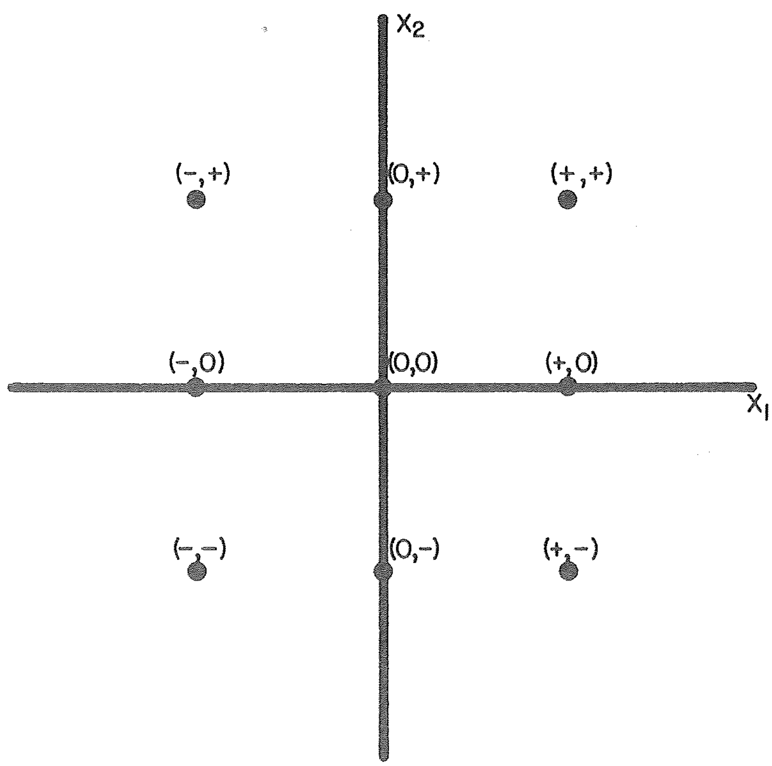
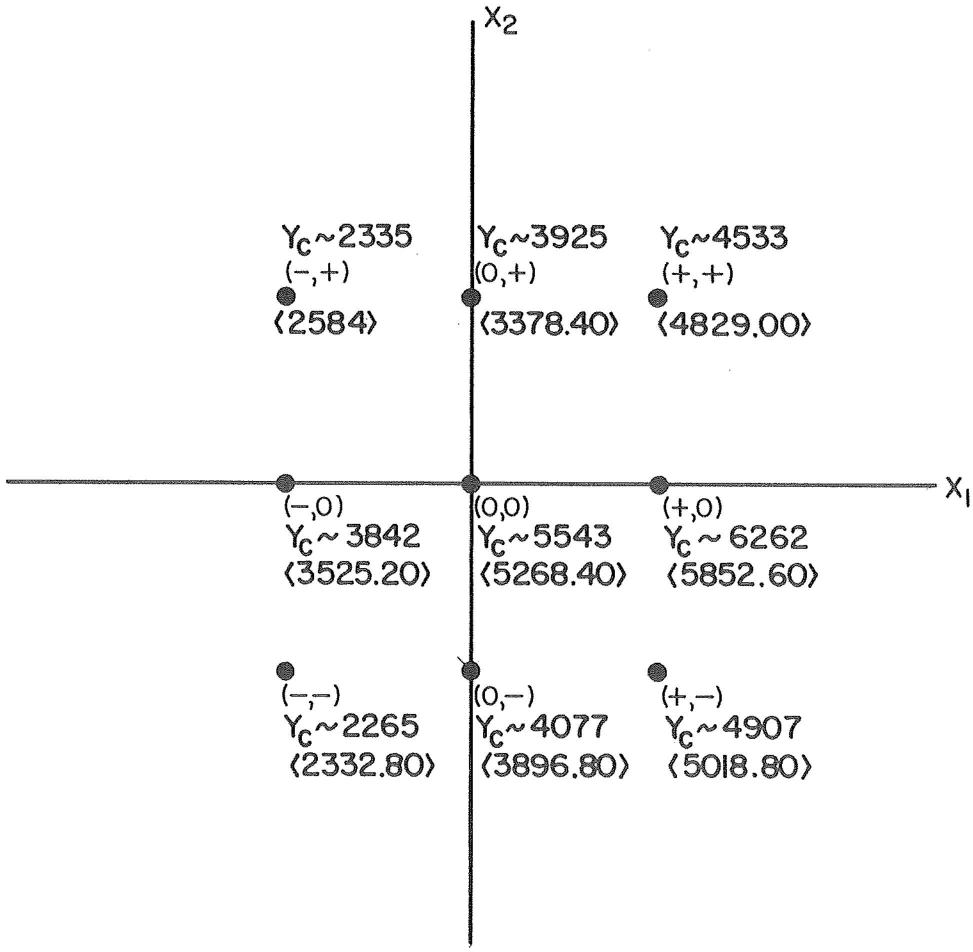


FIGURE 8

EMPIRICAL T,P EQUATION
with calculated values



$$Y = -491X_1^2 - 1542X_2^2 - 111X_1X_2 + 1210X_1 - 76X_2 + 5543$$

$$Y = f(X_1, X_2)$$

$$X_1 = \frac{P-120}{5} \qquad 115 \leq P \leq 125 \ (\tfrac{\text{lbs.}}{\text{ft.}^3})$$

$$X_2 = \frac{T-900}{200} \qquad 700 \leq T \leq 1100 \ (°C)$$

NOTE: to convert from pound-mass/ft.^{3} to kilogram/meter^3 (kg/m^3) mul. by 1.602 E+01

FIGURE 9

EXPERIMENTAL STD SURFACE

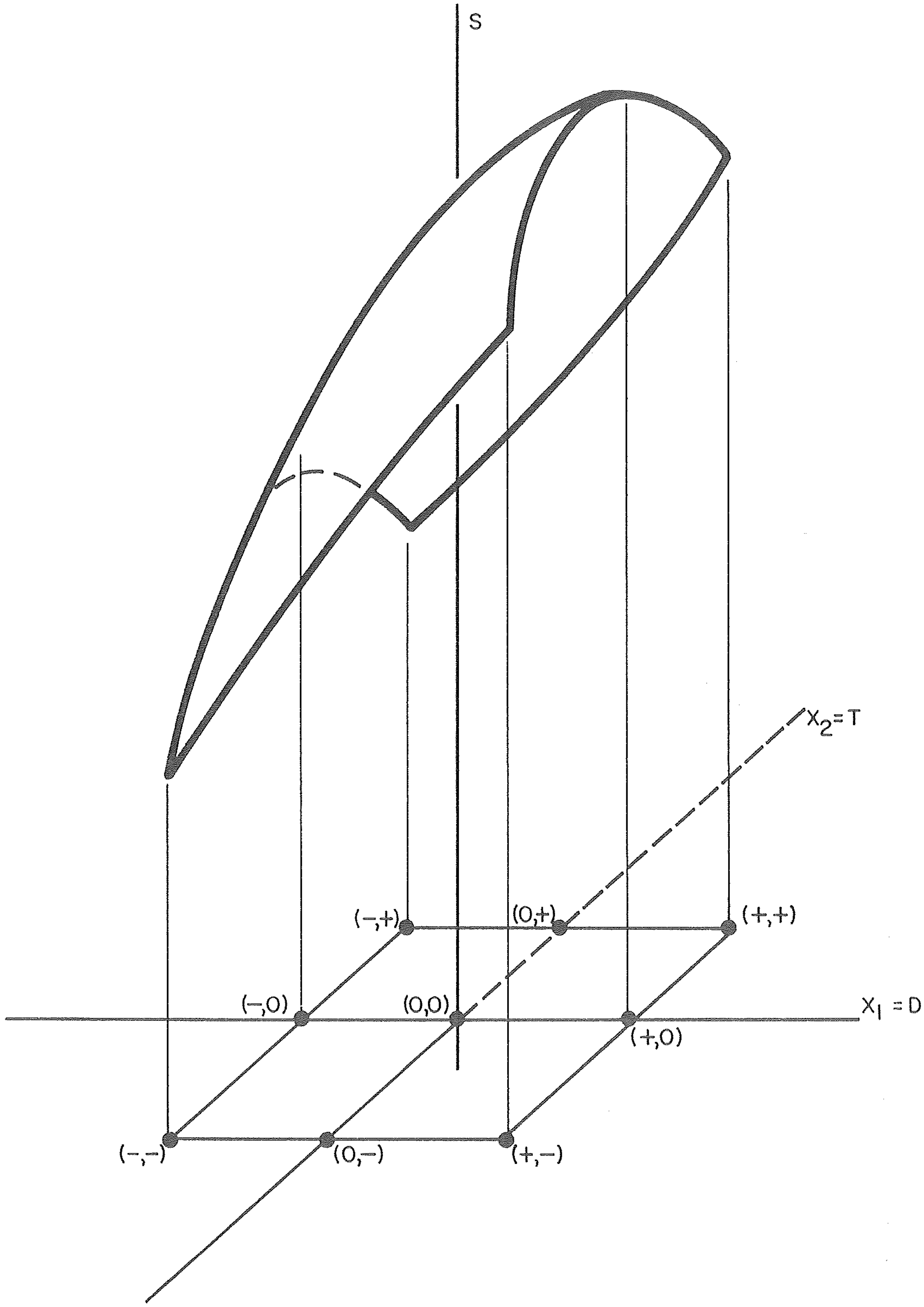


FIGURE 10

THEORETICAL STD SURFACE

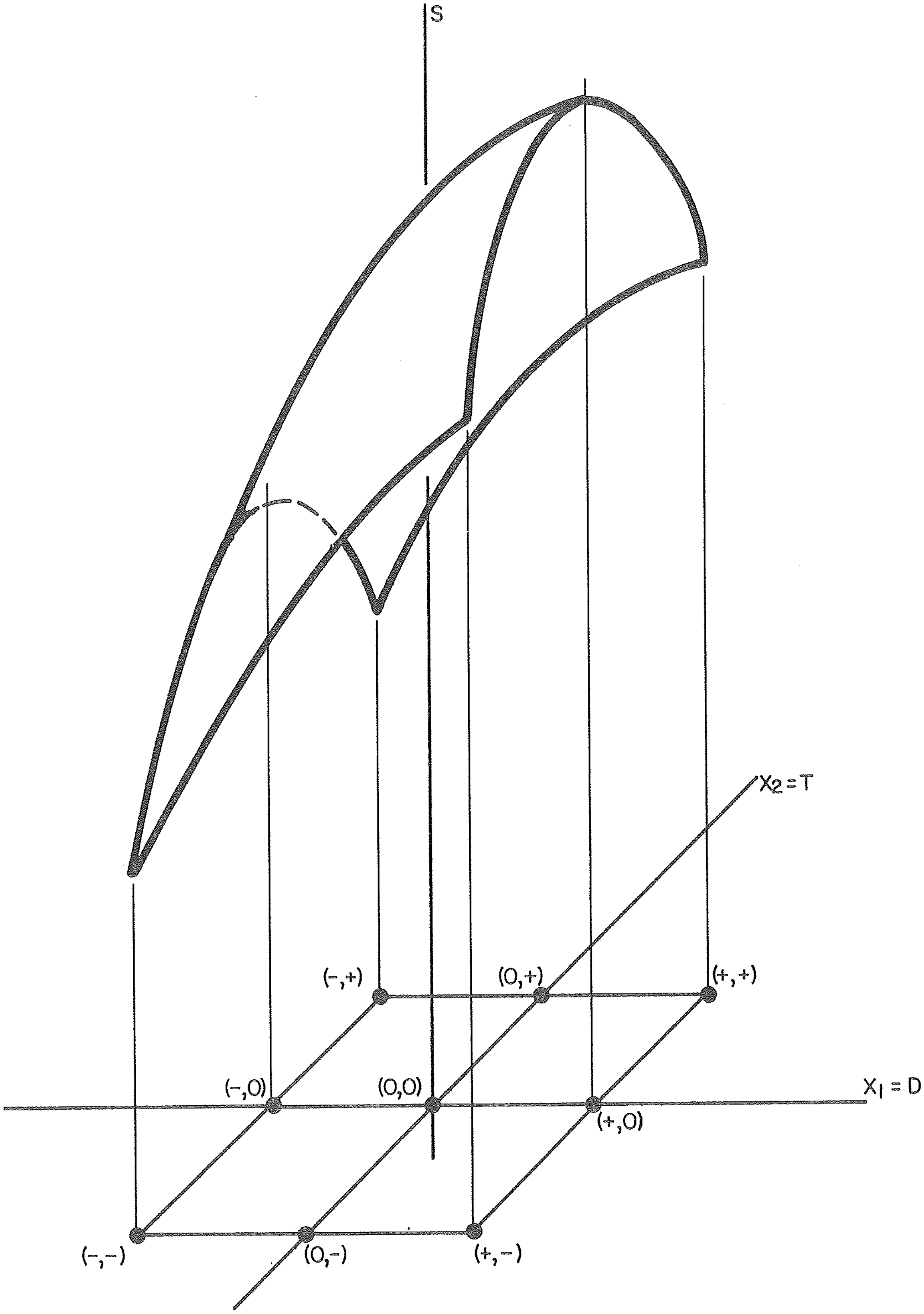


FIGURE 11

be produced. The engineering test results of this aggregate will be given in another section.

It should also be noted that the time of burning did not enter into the equation as a significant variable. Some light may be thrown on this apparent anomaly as follows. Since thermodynamic phase changes took place as the clay was heated to produce the aggregate, it is apparent that the period of time and range of temperature in which these phase changes occur was well within the time and temperature ranges chosen. Hence, if certain kinds of phase changes occurred completely within 20 minutes with a given temperature setting, then 15 or 20 minutes more firing time would not necessarily yield any additional change in the resulting product.

*NOTE: For the purpose of the production of the synthetic aggregate at T.T.I., these "Critical Values" were used as a guideline. It is recognized on the part of the researcher that it is risky business to differentiate a "fitted curve" and use the resulting critical points for further experimentation. However, based on the accumulated experience of the behavior of chinle clay samples under the sintering conditions used, these figures seemed to be good representative ones to employ as a general guidelines.

CHAPTER VI

FLY-ASH-CHINLE CLAY SYNTHETIC AGGREGATE

Synthetic aggregate produced with fly-ash additive was also investigated. From our previous work it was decided that two of the factors investigated would be temperature and density. Thus, we decided on a 3^3 factorial cubical design, see Figure12 . That is the levels were choses to be (–, 0,+). This would facilitate the analysis and enable a rapid fitting of the response surface. Sine $3^3 = (2 + 1)^3 = 2^3 + 3 \cdot 2^3 + 3 \cdot 2 + 1$, it was decided to obtain a "feel" for the main and interaction effects by initially considering a subset of the data as a 2^3 factorial design, see Figure13 .

It was found that all main effects were significant and all interaction terms were significant except the one between temperature and fly-ash content.

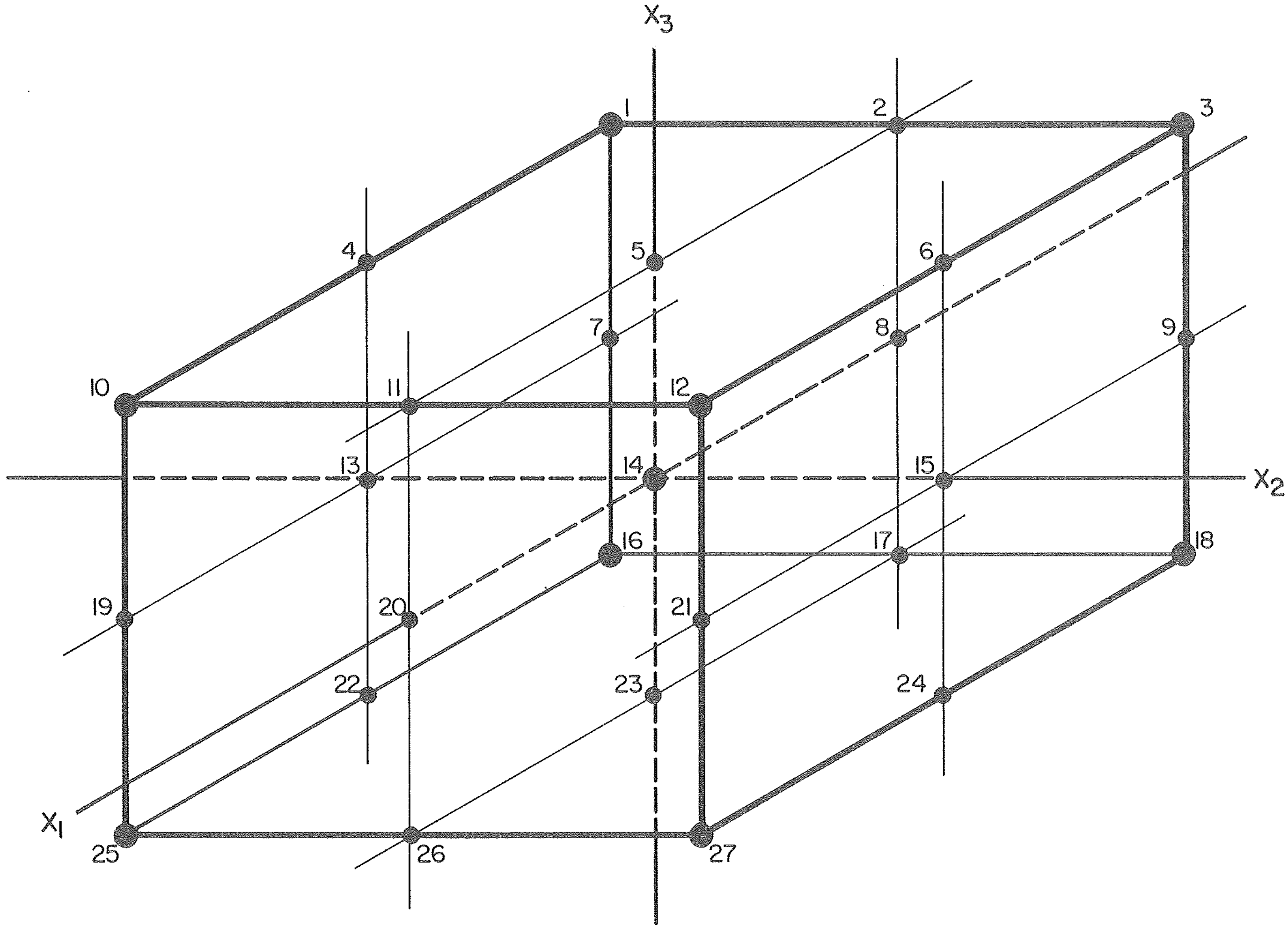
Moreover, the response surface was chosen to be a second degree equation; however, it was found that the second degree terms involving the main effects, temperature, fly-ash content and density, had to be dropped in order to obtain a reasonably good fit. This is due to the fact that throughout the selected range of the variables the response was dominated by a linear dependence on each of the main effects. It was found that including the significant interaction terms, i.e., temperature-denisty, fly-ash-density, temperature-fly ash-density, provided a fairly good fit.

The response equation was determined to be

$$y = 6231.28 - 1075.33x_1 - 619.70x_2 - 315.59\, x_3$$

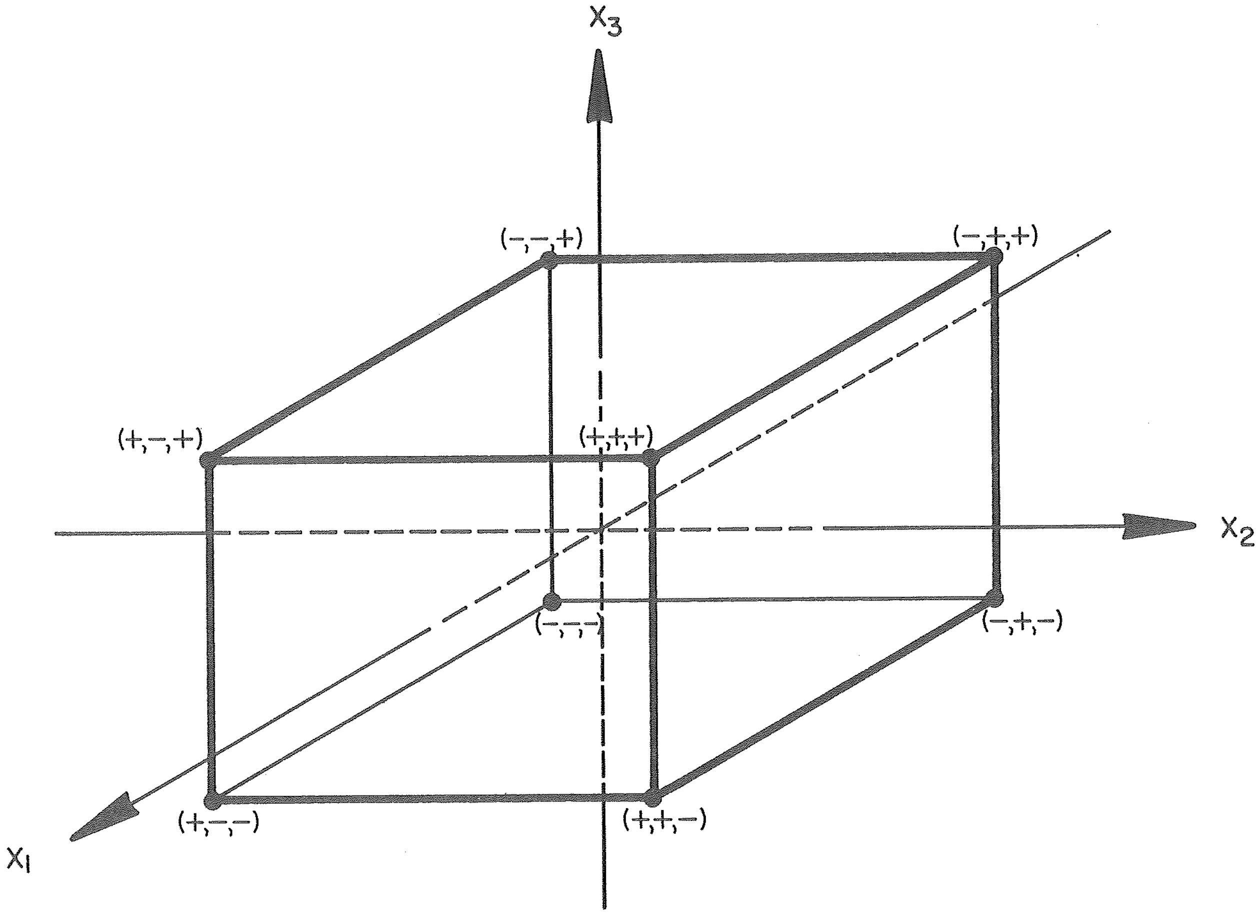
$$+ 429.28x_1\, x_3 - {}_3166.17x_2x_3 - 526.50x_1\, x_2\, x_3$$

GEOMETRIC DISPLAY OF 3^3 FACTORIAL



1. (−,−,+)	10. (+,−,+)	19. (+,0,0)
2. (−,0,+)	11. (+,0,+)	20. (+,0,0)
3. (−,+,+)	12. (+,+,+)	21. (+,0,+)
4. (0,−,+)	13. (0,−,0)	22. (0,−,−)
5. (0,0,+)	14. (0,0,0)	23. (0,0,−)
6. (0,+,+)	15. (0,+,0)	24. (0,+,−)
7. (−,−,0)	16. (−,−,−)	25. (+,−,−)
8. (−,0,0)	17. (−,0,−)	26. (+,0,−)
9. (−,0,+)	18. (−,+,−)	27. (+,+,−)

FIGURE 12



GEOMETRIC DISPLAY
OF 2^3 FACTORIAL

FIGURE 13

where $x_1 = \dfrac{T-1000}{200}$, $x_2 = \dfrac{CFA-10}{5}$ and $x_3 = \dfrac{D-100}{10}$

This indicates that increasing the temperature, fly-ash content and density would generally bring a reduction in strength. However, it should be noted that the interaction term (x_1 x_3) has a positive coefficient which indicates that the temperature and density when increased simultaneously act to strengthen the material as expected.

The experimental data showed wide scatter when the three-replications were compared. Consequently, the equation gave some predicted values which were about ±20% different than the replication average value, but if the relatively wide scatter in the experimental data is taken into account and the fact that overall our equation gives an average signed error of only +1.78%, it is reasonable to fully utilize the equation throughout the chosen range of temperature, fly-ash content and wet density.

The next set of experiments reported on are those concerning attempts a producing a synthetic aggregate using chinle clay and Winslow blow sand.

CHAPTER VII

WINSLOW BLOW SAND & CHINLE CLAY SYNTHETIC AGGREGATE

During the course of this project a limited series of experiments was carried out to determine the value of using chinle clay and blow sand* together as possible materials for producing synthetic aggregate.

LABORATORY PROCEDURE USED

All test samples were designed for manual compaction to a cylinder 1.25" high x 1.25" diameter (3.17 cm high x 3.17 cm dia.)

The post ignition samples were not capped with Cylcap prior to the compressive strength test because of the fact that the Cylcap cap changes the chemical composition of this small sample, and the Cylcap also interferes with specific gravity tests on this small sample size.

Two sample batches were manually mixed and stored in plastic bags, placed inside of concrete cylinder cans. The bags were tied and the cylinder cans sealed with tape.

Each batch contained sufficient material for 70 samples, although only 60 samples were used per batch. The remaining material has been sealed and retained.

Date of Making:	November 19, 1973
Aged in plastic bags until:	November 27, 1973
Weighed and compacted all molds:	#358A - #363F - November 27, 1973 (Sealed inside tared tin cans against moisture changes). Stored in cool area.
	#364A - #377F - November 3, 1973

*aeolian deposited material with a low shear angle.

All molds air dried: December 3, 1973 - December 7, 1973

All molds oven dried in Blue M Electric oven @ 105°C from December 7, 1973 to December 10, 1973.

1st Burn - December 11, 1973
400°C through 800°C

2nd Burn - December 12, 1973
900°C through 1300°C

Uncapped compressive strength testing:

#358A - #361F - December 13, 1973
#362A - #377F - December 14, 1973

All samples stored in their original tared tin cans to await further testing, if any.

MATERIALS USED

In this short series of experiments the following materials were used:

Holbrook Chinle Clay:

Lab #72-4054

No. -40" material at equilibrium moisture.
(5.1638 wt-% Initial Moisture - 105°C/24 hrs.)

Winslow Blow Sand:

Lab #73-9089

Equilibrium moisture and no pre-treatment.)
1.0811 wt-% Initial moisture, 105°C/24 hrs.)

The following factors were used at the indicated levels:

S_1 = Winslow Blow Sand - 25 wt %
Holbrook Chinle Clay - 75 wt %

S_2 = Winslow Blow Sand - 50 wt %
Holbrook Chinle Clay - 50 wt %

t_1 = 15 minutes at nominal temperature

t_2 = 35 minutes at nominal temperature

P_1 (wet) = P_{w1} = 128.7#/ft^3

P_2 (wet) = P_{w2} = 138.6#/ft^3

M_1 (H20) = Moisture content M_{c1} = 11.3 wt %

M_2 (H20) = Moisture content = M_{c2} = 13.9 wt %

T_n°C = Nominal temperature, where
T = 400°C through 1300°C.

EXPERIMENTAL OUTLINE USED FOR
HOLBROOK CHINLE CLAY AND WINSLOW BLOW SAND

SAMPLE TESTING OUTLINE

SAMPLE #	TEMP °C		FIRING TIME 15 MIN.	35 MIN.	WET UNIT WEIGHT, GMS.
358	400 --	S_1	15 min.	35 min.	(57.7)
359		S_2	15 min.	35 min.	(55.8)
360	500 --	S_1	15 min.	35 min.	57.7
361		S_2	15 min.	35 min.	55.8
362	600 --	S_1	15 min.	35 min.	57.7
363		S_2	15 min.	35 min.	55.8
364	700 --	S_1	15 min.	35 min.	57.7
365		S_2	15 min.	35 min.	55.8
366	800 --	S_1	15 min.	35 min.	57.7
367		S_2	15 min.	35 min.	55.8
368	900 --	S_1	15 min.	35 min.	57.7
369		S_2	15 min.	35 min.	55.8
370	1000 --	S_1	15 min.	35 min.	57.7
371		S_2	15 min.	35 min.	55.8
372	1100 --	S_1	15 min.	35 min.	57.7
373		S_2	15 min.	35 min.	55.8
374	1200 --	S_1	15 min.	35 min.	57.7
375		S	15 min.	35 min.	55.8
376	1300 --	S_1	15 min.	35 min.	57.7
377		S_2	15 min.	35 min.	55.8

EMPIRICAL EQUATION OF STATE

After the data was obtained it was submitted to a non-linear regression analysis and the following empirical equation of state was obtained.

$$\text{Log } x_5 = 4.61 + .087 \text{ Log } y + .073 \text{ Log } x_1 + 0.13 \text{ Log } x_2 + 0.26 \text{ Log } x_3 + 0.29 \text{ Log } x_4$$

WHERE

x_1 = Mc (wt-% water on dry soil basis) for wet molds

x_2 = Time in minutes at temperature, $T_n\,^{o}C$

x_3 = P_{wet} (density, in pounds/foot^3, of wet mold)

x_4 = wt-% Winslow blow sand

x_5 = Compressive strength of uncapped mold

y = $T_n\,^{o}C$ for time t_n

Comparison of compressive strength values obtained from laboratory testing and calculated from empirical equation of state.

CALCULATED	EXPERIMENTAL
3060.88 PSI	3060.95 PSI

CHAPTER VIII

COMMERCIAL SCALE PRODUCTION

Based on the results of laboratory production of synthetic aggregate it was decided to produce a large volume of aggregate using commercial-like techniques. Approximately 6 cubic yards of chinle clay was sent to the Texas Transportation Institute (T.T.I.) for sintering into synthetic aggregate. The clay was fired at temperatures ranging from 760°C (1400°F) to 1100°C (1900°F) and the resulting aggregate was returned to the Arizona Department of Transportation's Materials Services for testing and evaluation. Due to the shortness of remaining testing time it was decided to only test the aggregate fired at 1100°C (1900°F). This decision was partially based on the fact that the aggregate fired at 1100°C (1900°F) had not broken up during shipment back to A.D.O.T. as much of the aggregate fired at the lower temperatures had. Moreover the aggregate fired at 1100°C (1900°F) appeared, under visual examination, to have undergone more recrystallization.

The laboratory tests on the synthetic aggregate consisted of the following:

(1) Bituminous
(2) Concrete
(3) Aggregate Evaluation

BITUMINOUS MIXES

The synthetic aggregate was used in the following tests:

(1) Bitumen percentage
(2) Effect of water on compacted bituminous mixes
(3) Stability and cohesion

(4) Rice tests

(5) C.K.E.

For results of these tests, see Appendix A.

BITUMEN TEST CONCLUSIONS

Based on the above tests, the Arizona Department of Transportation Materials Services has concluded that the aggregate as received from T.T.I. is of marginal quality. The asphalt absorption is too high for good economics, but the compatibility with asphalt as demonstrated by the immersion-compression test is good.

CONCRETE SECTION

The synthetic aggregate was used in the following tests:

(1) Concrete mix design (Class A)

(2) Duplicate cylinder breaks at 7 and 28 days

(3) Beam break at 7 days

For the results of these tests, see Appendix A.

CONCRETE TEST CONSLUSIONS

The results of the concrete tests indicate that a 1750 psi 7-day concrete can be obtained using a standard mix and the sythetic aggregate. In addition, the 7-day beam break gave an MR of 300.

The final 28-day test data comparing the synthetic aggregate to a Salt River aggregate in a concrete mix is given below:

SALT RIVER AGGREGATE		SYNTHETIC AGGREGATE	
LOAD	STRESS	LOAD	STRESS
127,500 LBS.	4,510 psi	107,500 LBS.	3,800 psi
126,500 LBS.	4,470 psi	105,000 LBS	3,710 psi
*57,885 kg	3.11E+07 Pa	48,805 kg	2.62E+07 Pa
*57,431 kg	3.06E+07 Pa	47,610 kg	2.56E+07 Pa

*S.I. UNITS.

Thus, based on the concrete cylinder test the aggregate performed approximately as well as standard Salt River aggregate and, therefore, may be used as a construction material in the production of standard weight concrete.

AGGREGATE SECTION

The synthetic aggregate was used in the following tests:

(1) Crush samples and determine gradation

(2) Abrasion tests

(3) Soundness-sodium sulfate method

(4) Specific gravity and absorption

(5) Sand equivalent test

For the results of these tests, see Appendix A.

AGGREGATE SECTION CONSLUSIONS

Based on the abrasion tests the aggregate fired at 1100°C (1900°F) had approimately 13% loss at 100 revolutions and 47% loss at 500 revolutions. The specific gravity was approximately 2.30* and the material was non-plastic. Based on these results, the material appears to be suitable for certain phases of highway construction; however, it is suggested that further tests be performed to verify this conclusion.

It is stressed that due to the shortness of remaining testing time only the aggregate fired at 1100°C was thoroughly tested.

*For the coarse aggregate, the fine aggregate had a sp. gr. of 2.50.

CHAPTER IX

CONCLUSIONS AND RECOMMENDATIONS

Based on our laboratory studies and the commercial production of synthetic aggregate by the Texas Transportation Institute the following conclusions have been reached.

(1) A suitable synthetic aggregate can be produced from chinle clay when produced at a temperature of 1100°C (1900°F). The aggregate fired at 1100°C (1900°F) was the only sample thoroughly tested because of the shortness of testing time hence the same statement cannot be made regarding the aggregate produced at the lower temperatures.

(2) The density of the pellets was found to be one of the most significant factors influencing the production of a suitable aggregate. It is suggested to start with pellets that have a wet density in the range of 115 lbs/ft^3 (1840 kg/m^3) to 125 lbs/ft^3 (2000 kg/m^3).*

(3) The length of sintering time is not critical and, therefore, should be held to a minimum for economic reasons. It is recommended that 20 minutes be used as a guideline.

(4) Mixing moisture content had minimal influence on the final product, therefore, it is suggested that sufficient water be mixed with the green clay to make the mixture workable.

(5) The clay content was not found to be a significant main effect over the range of 24 to 40%. This means that even though there is a wide range of clay content in the naturally occurring material, it can be used to produce a uniform synthetic aggregate.

*See Page 34

(6) The asphalt tests on the T.T.I. aggregate indicated that the material has possible uses as a highway material.

(7) The concrete tests indicate that a suitable strength can be obtained using this aggregate.

RECOMMENDATIONS

The following recommendations are based on this study.

(1) Further tests be conducted on the T.T.I. aggregate produces at temperatures below 1100°C (1900°F).

(2) Evaluate the suitability of this aggregate as a highway material.

(3) Investigate the possibility of local contractors producing synthetic aggregate using existing equipment.

(4) Determine the economics of the production of synthetic aggregate produced by local contractors.

(5) Since all commercial production was performed on pelletized material, it is suggested that a 2 cubic yard of +1 inch material be sent to T.T.I. for the production of synthetic aggregate from this size material.

(6) Test and evaluate the aggregate produced from recommendation Number 5.

IMPLEMENTATION STATEMENT

It is the feeling of the operational branch which conducted the research that the Arizona Department of Transportation is in a position to initiate selected activities as a result of the findings of this research. The proper timing to implement these activities will be dependent on future management considerations.

Because of the lack of suitable natural aggregate in Northwestern Arizona it is vital that the Arizona Department of Transportation have some information on producing aggregate from natural materials available in that region of Arizona. This report brings together a large volume of such information and data for producing synthetic aggregate from expansive clays for future contingencies.

COST ESTIMATE FOR PRODUCTION OF SYNTHETIC AGGREGATE

It has been estimated that synthetic aggregate could be produced from Chinle Clay at a cost of $2/ton. This figure is based on current costs to aggregate producers using materials similar to that of Chinle Clay and processes similar to the one used to produce this synthetic aggregate.

APPENDIX A

LABORATORY RESULTS OF PHYSICAL TESTING

On the following pages are the results of the test procedures used in evaluating the synthetic aggregate.

APPENDIX A

TABLE 4

ADOT - MATERIALS SERVICES

LABORATORY BITUMINOUS MIXTURE DESIGN
SYNTHETIC AGGREGATE

SIEVE SIZE	% RETAINED	% PASSED
1/2"	0	100
3/8"	26	74
1/4"	11	63
#4	7	56
#8	18	38
#10	5	33
#16	2	31
#30	11	20
#40	14	6
#50	2	4
#100	2	2
#200	1	1

TEST RESULTS	COARSE AGGREGATE	FINE AGGREGATE	COMBINED AGGREGATE
S.G.	2.295	2.489	2.382
Absorption	8.18%	3.29%	5.74%
O.D.S.G.	2.120	2.410	2.282

TABLE 4

LABORATORY BITUMINOUS MIXTURE DESIGN (Cont'd.)

DESIGN DATA

Specimen	A	B	C	D	E	F	Design Spec.	
Bulk Density lbs/ft^3	118.1	121.8	120.6	123.5	123.2	124.3	124.7	124.9
Bit. Grade	AR-4000							
% Bit.	6.0	6.5	7.8	8.0	9.0	10.0	10.6	11.0
Density, lbs per cu. ft.	114	115	116	117	116	123		122
Stabilometer	36	33	35	42	41	40		40
Cohesiometer	23	28	30	37	43	57		136
Voids						7.5	6.8	6.0
V.M.A.						21.4	21.8	22.6
% Voids Filled						65.0	68.8	72.7
Effective Asphalt, Total Mix						7.06	7.68	8.09

Sample	Air PSI	H_2O PSI	Retention	
No. 1	183	183	100%	10.6% AR-4000 Asphalt
No. 2	183	198	108%	1.0% Anti Strip Agent
No. 3			%	% Dry Lime
No. 4	183	203	111%	2.0% Dry Cement

Max. Density 133.8 lbs per cu. ft. (Rice Method) 10.6%

Asphalt 3.27% Absorption on Dry Aggregate

C.K.E. Values F = 3.86 C - 6.86

Theoretical Bit. Ratio For Grade: AR-4000 Bitumen 6.1 %

K_c = 2.8 K_f = 1.6 K_m = 2.0

NOTE: for conversion to S.I. units see page 58.

TABLE 5

CONCRETE MIXES AND TEST RESULTS

	Salt River Mix	Synthetic Aggregate Mix
Slump (in)	1.5	1
Air Content (%)	2	3
7-Day Compressive Strength (psi)	2520	1750
28-Day Compressive Strength (psi)	4490	3760
7-Day Beam Break Modulus of Rupture (psi)	400	300
Cement (lbs.)	564	564
Synthetic Aggregate (lbs.)	---	2760
Salt River Fine Aggregate (lbs.)	1756	---
Salt River Coarse Aggregate (lbs.)	1375	---
Water (gals.)	36	45

Concrete mixes based on S.S.D. Design Weights

TO CONVERT FROM	TO	MUL.BY
pound-mass (avoirdupois)	kilogram	4.535924 E-01
pound-force/inch^2 (psi)	pascal (Pa)	6.894757 E+03
gallon (U.S. liquid)	litre	3.785412 E+00
inch	meter (m)	2.540000 E-02
pound-mass/foot^3	kilogram/meter^3	1.601846 E+01

Table 6

Soil and Aggregate Properties

Sample #	% Pass ½" Sieve	% Pass 3/8" Sieve	% Pass #4 Sieve	% Pass #40 Sieve	% Pass #200 Sieve	P.I.	Coarse Agg. Spec. Gravity	Coarse Agg. Abs.(%)	Fine Agg. Spec. Gravity	Fine Agg. Abs. (%)	Sand Equiv.	Type "C" 100 Rev %	Abrasion 500 Rev %
AHD-1 1790^0F & 56 Min. Retention	100	58	32	9	3	N.P.	2.19	10.6	--	--	88	16	55
AHD-2 1900^0F & 56 Min. Retention	100	44	36	0	0	N.P.	--	--	--	--	100	--	--
AHD-3 1900^0F & 41 Min. Retention	100	62	45	6	1	N.P.	2.29	7.2	--	--	98	13	46
AHD-4 1800^0 & 41 Min. Retention	100	70	52	5	1	N.P.	2.18	10.8	--	--	95	15	52
AHD-5 1700^0F & 41 Min. Retention	100	76	57	9	2	N.P.	2.17	11.4	--	--	93	14	49
AHD-6 1700^0F & 56 Min. Retention	100	73	54	6	1	N.P.	2.13	12.2	--	--	91	16	55
AHD-2Crushed 1900^0F & 56 Min. Retention	100	65	52	10	3	N.P.	2.32	7.9	2.50	5.8	92	13	47

* Sample used for Asphaltic Concrete Mix Design and Concrete Mix Design
Sodium Sulfate Soundness: 5 Alternations 1% on Fine and Coarse Aggregate

TABLE 6

SOIL AND AGGREGATE PROPERTIES

SAMPLE	% PASS 1/2" SIEVE	% PASS 3/8" SIEVE	% PASS #4 SIEVE	% PASS #40 SIEVE	% PASS #200 SIEVE	P.I.	COARSE AGG. SPEC. GRAVITY	COARSE AGG. ABS. (%)	FINE AGG. SPEC. GRAVITY	FINE AGG. ABS (%)	SAND EQUIV.	TYPE "C" 100 REV %	ABRASION 500 REV %
AHD-1 1790°F & 56 Min. Retention	100	58	32	9	3	N.P.	2.19	10.6	-----	---	88	16	55
AHD-2 1900°F & 56 Min. Retention	100	44	36	0	0	N.P.	----	----	-----	---	100	--	--

TABLE 7

WT-% OXIDE ANALYSIS OF USP BENTONITE

	Wt-% OXIDE
Si	58.4
Al	20.8
Fe	3.04
Mn	0.0
Mg	1.95
Ca	.67
Sr	.08
Na	1.04
K	.48
Li	-----
Ga	.05
Zn	.037
Cr	.01
Cu	.15
Ni	.34
V	0.0
Ti	.07

TABLE 8

WT-% OXIDE ANALYSIS OF WYOMING BENTONITE

	Wt-% OXIDE
Si	57.9
Al	19.6
Fe	4.65
Mn	.11
Mg	2.19
Ca	1.14
Sr	.08
Na	.776
K	.59
Li	-----
Ga	.04
Zn	.02
Cr	.01
Cu	.048
Ni	-----
V	0.0
Ti	.13

TABLE 9

WT-% OXIDE ANALYSIS OF
HOLBROOK TEST SITE CHINLE CLAY SAMPLE

	Wt-% OXIDE
Si	58.5
Al	20.1
Fe	4.85
Mn	.055
Mg	2.13
Ca	.31
Sr	.07
Na	1.01
K	1.26
Li	-----
Ga	.01
Zn	.14
Cr	.02
Cu	.68
Ni	1.12
V	0.0
Ti	.60

TABLE 10

WT-% OXIDE ANALYSIS OF
HOLBROOK CHINLE CLAY SAMPLE AND
CORES FROM SYNTHETIC AGGREGATE SAMPLES

	HOLBROOK CHINLE CLAY	CORE 188 900°C FOR 30 MIN.	CORE 189 1000°C FOR 30 MIN.	CORE 193 1100°C FOR 30 MIN.
Si	54.92	59.06	60.73	61.94
Al	19.31	22.3	23.41	24.09
Fe	4.87	6.01	6.02	6.13
Mn	.048	-----	-----	-----
Mg	1.69	1.40	1.44	1.50
Ca	.80	.778	.781	.786
Sr	.01	-----	------	------
Na	.39	.415	.425	.437
K	.33	.307	.308	.34
Li	----	-----	-----	-----
Ga	0.0	-----	-----	-----
Zn	.082	-----	-----	-----
Cr	.0095	-----	-----	-----
Cu	.13	-----	-----	-----
Ni	.23	-----	-----	-----
V	0.0	-----	-----	-----
Ti	.94	1.01	1.12	1.14

TABLE 11

HOLBROOK 1100^{o}C @ 30 MIN. SYNTHETIC AGGREGATE CORE VS US 89, MP 489.4 1100^{o}C @ 30 MIN. SYNTHETIC AGGREGATE CORE (PEGC-4R) WT-% OXIDE

	HOLBROOK CORE 193	US 89 CORE
Si	61.74	59.2
Al	24.09	22.09
Fe	6.13	6.55
Mn	-----	.037
Mg	1.50	1.13
Ca	.786	1.08
Sr	-----	.042
Na	.437	.353
K	.34	2.93
Li	-----	-----
Ga	-----	.046
Zn	-----	.052
Cr	-----	.0028
Cu	-----	.26
Ni	-----	.50
V	-----	0.0
Ti	1.14	.71

TABLE 12

WT-% OXIDE ANALYSIS OF
ARIZONA PUBLIC SERVICE COMPANY FLY ASH

	WT-OXIDES
Si	54.5
Al	24.9
Fe	3.57
Mn	.046
Mg	.91
Ca	3.6
Sr	.04
Na	.51
K	.87
Li	-----
Ga	.29
Zn	.027
Cr	0.0
Cu	.078
Ni	.12
V	-----
Ti	.84

DTA THERMOGRAPHS

Several different materials were subjected to DTA, they were: Kaolinite, Wyoming Bentonite, USP Bentonite, Calcite, Chinle Clay and Arizona Public Service fly ash.

The heating rate employed was $10^{o}C/min.$ $(50^{o}F/min)$ with alumina used as the inert reference material.

All of the thermographs show a preliminary endothermic reaction occurring in the range of $70^{o}C-85^{o}C$ which is the removal of absorbed water. Moreover, there is a deflection in the graphs of Figures 1-5 at temperatures of about $600^{o}C-800^{o}C$. This is an endothermic deflection indicating the removal of lattice water. Note that the bentonite material's deflection occurs at a higher temperature $(720^{o}C)$ than the corresponding reaction for the Kaolin $(592^{o}C)$.

In Figure 4 the large endothermic deflection, at $975^{o}C$, is thought to be the destruction of the calcite lattice.

The "chinle clay" thermograph shown in Figure 5 shows a slight deflection at $920^{o}C$, this is probably due to the formation of spinel.

The APS fly ash behaved more like an inert material after a temperature of $310^{o}C$ was reached.

An inert atmosphere of nitrogen was used to prevent the occurrence of interfering thermaloxidation effects of organic materials present in the sample.

SAMPLE: KAOLIN
TEMP. RANGE: AMBIENT TO 1200°c
ATMOSPHERE: N_2

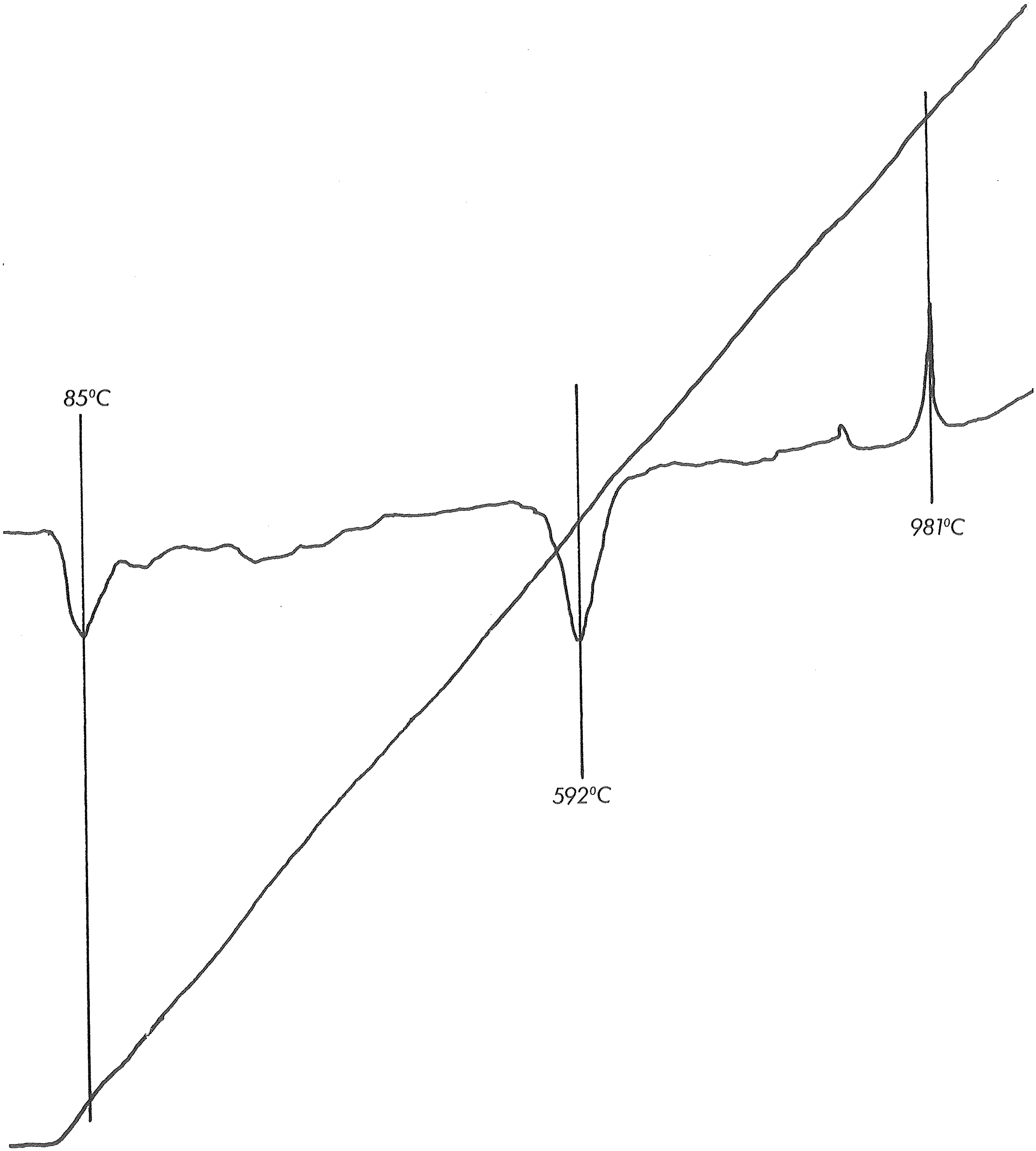


FIG. 1

SAMPLE: WYOMING BENTONITE
TEMP. RANGE: AMBIENT TO 1200°c
ATMOSPHERE: N_2

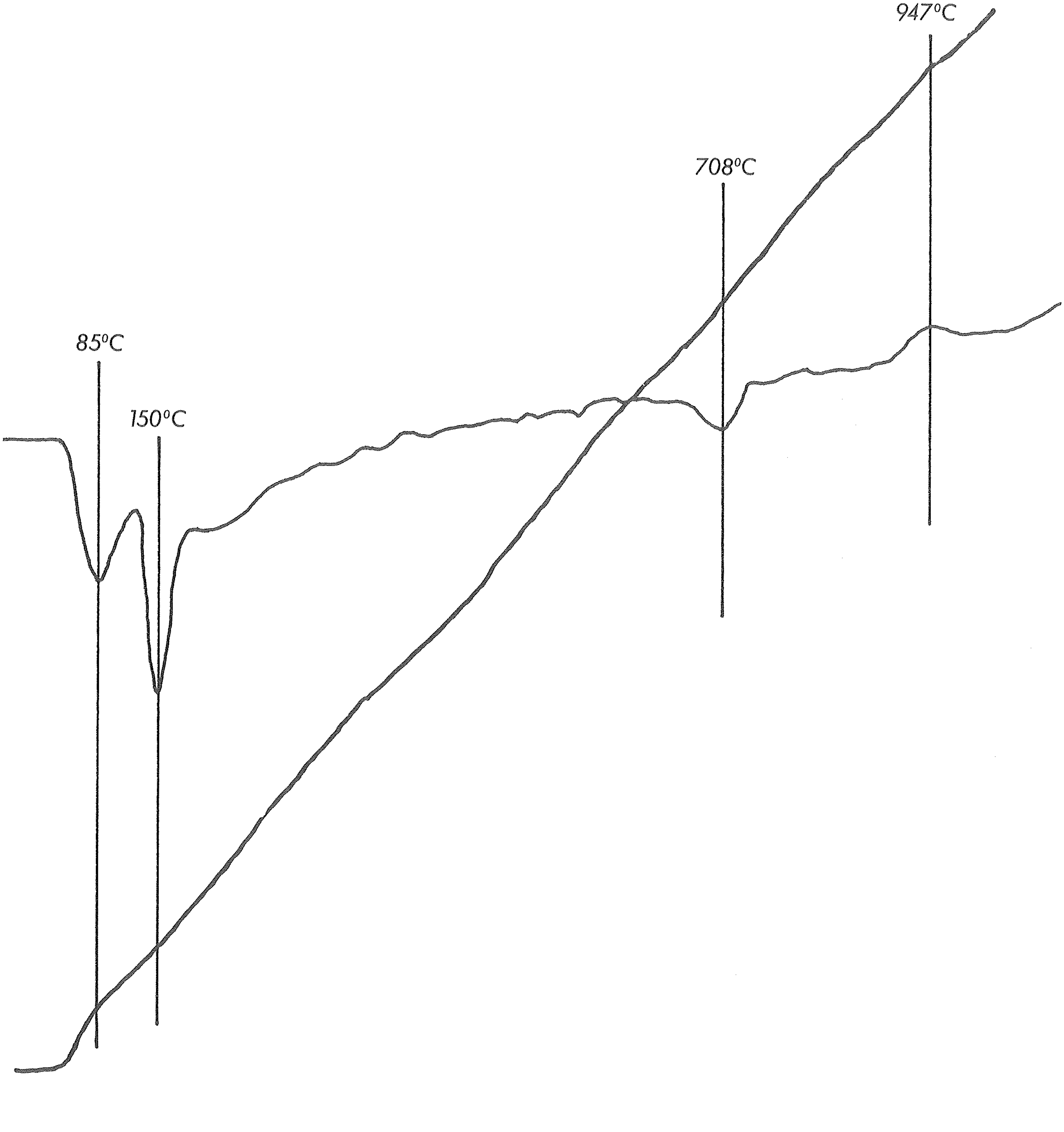


FIG. 2

SAMPLE: USP BENTONITE
TEMP. RANGE: AMBIENT TO 1200°c
ATMOSPHERE: N_2

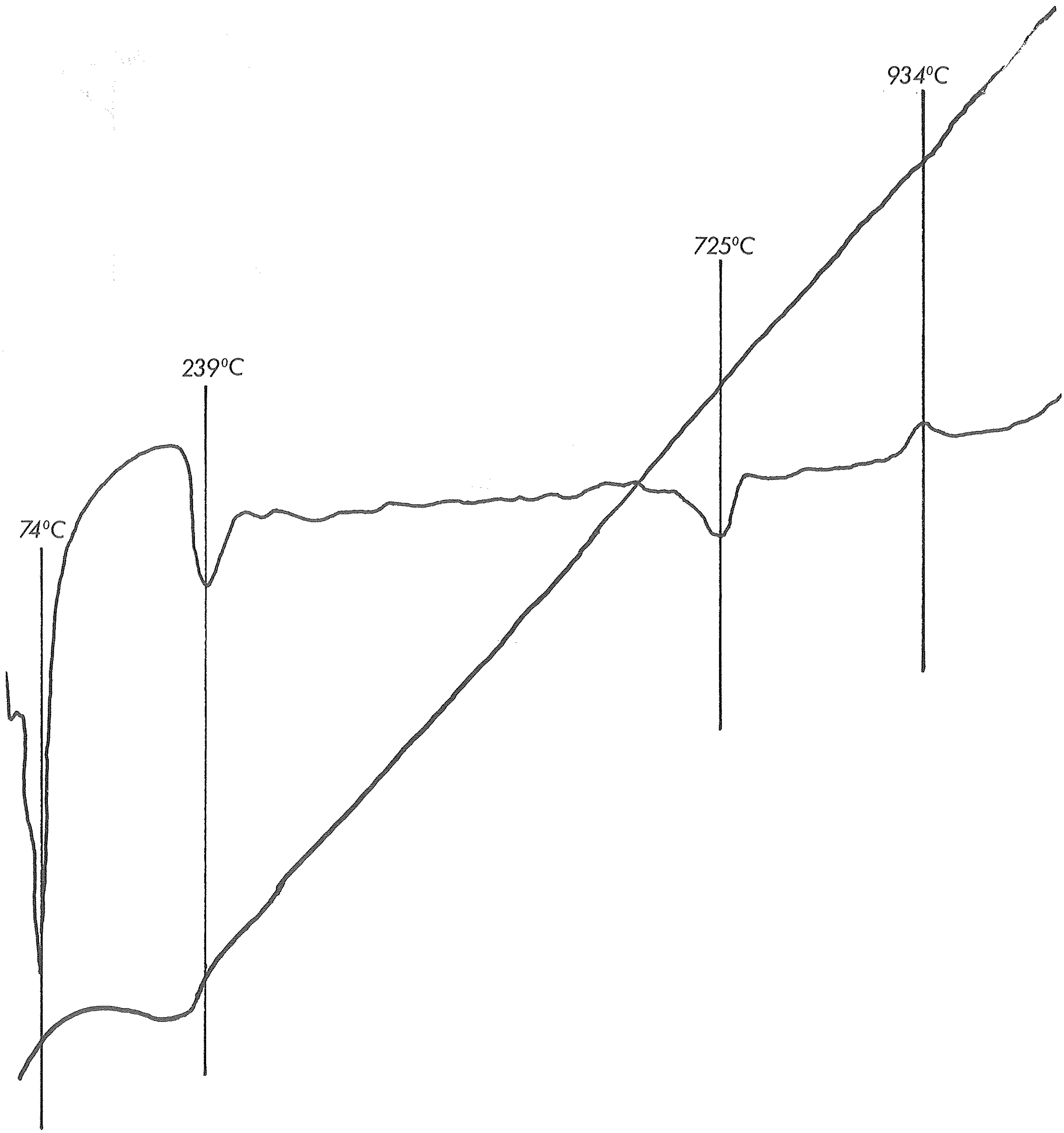


FIG. 3

SAMPLE: CALCITE
TEMP. RANGE: AMBIENT TO 1200°c
ATMOSPHERE: N_2

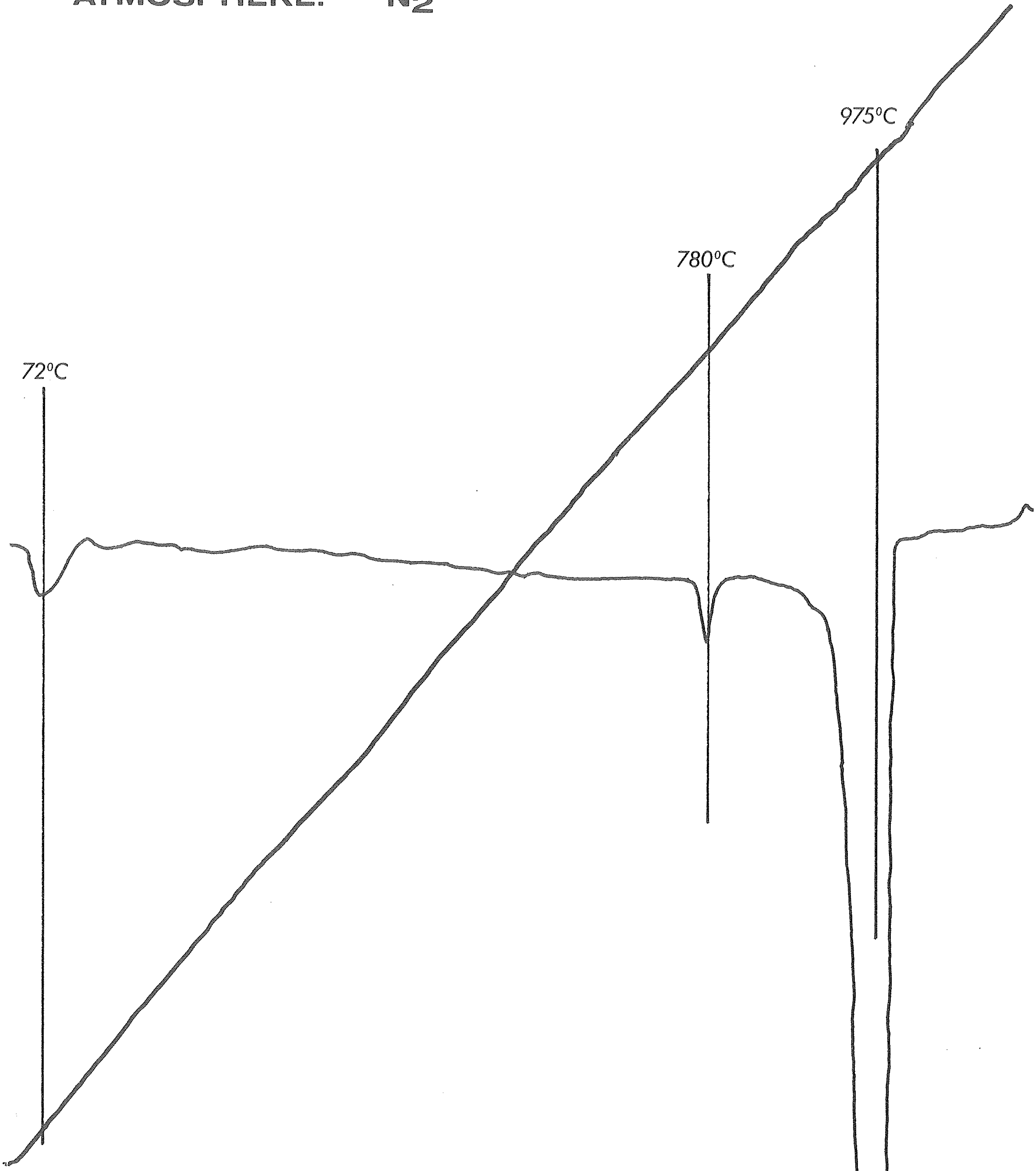


FIG. 4

SAMPLE: CHINLE CLAY
TEMP. RANGE: AMBIENT TO 1200°c
ATMOSPHERE: N_2

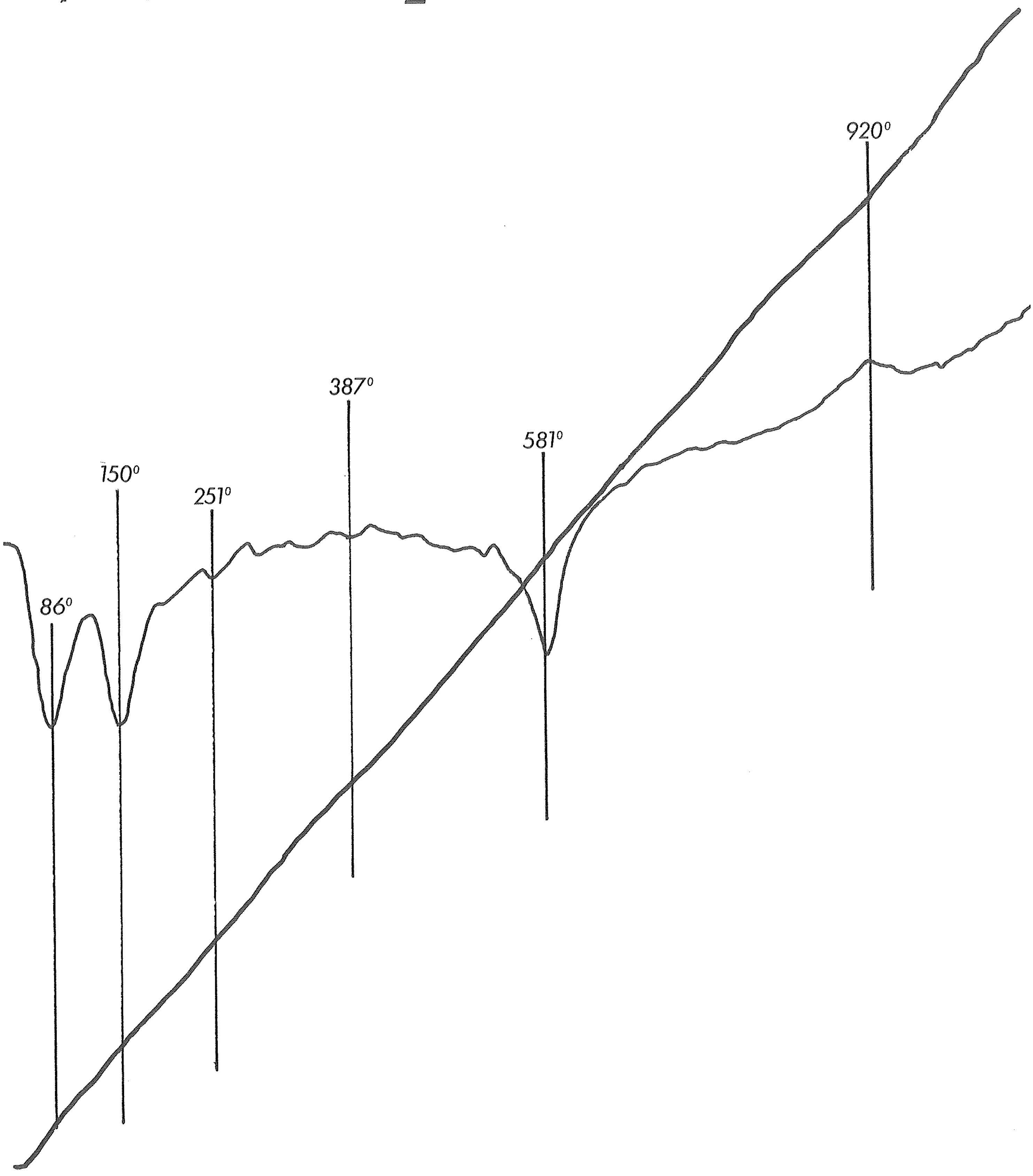


FIG. 5

SAMPLE: APS FLY ASH
TEMP. RANGE: AMBIENT TO 1200°c
ATMOSPHERE: N_2

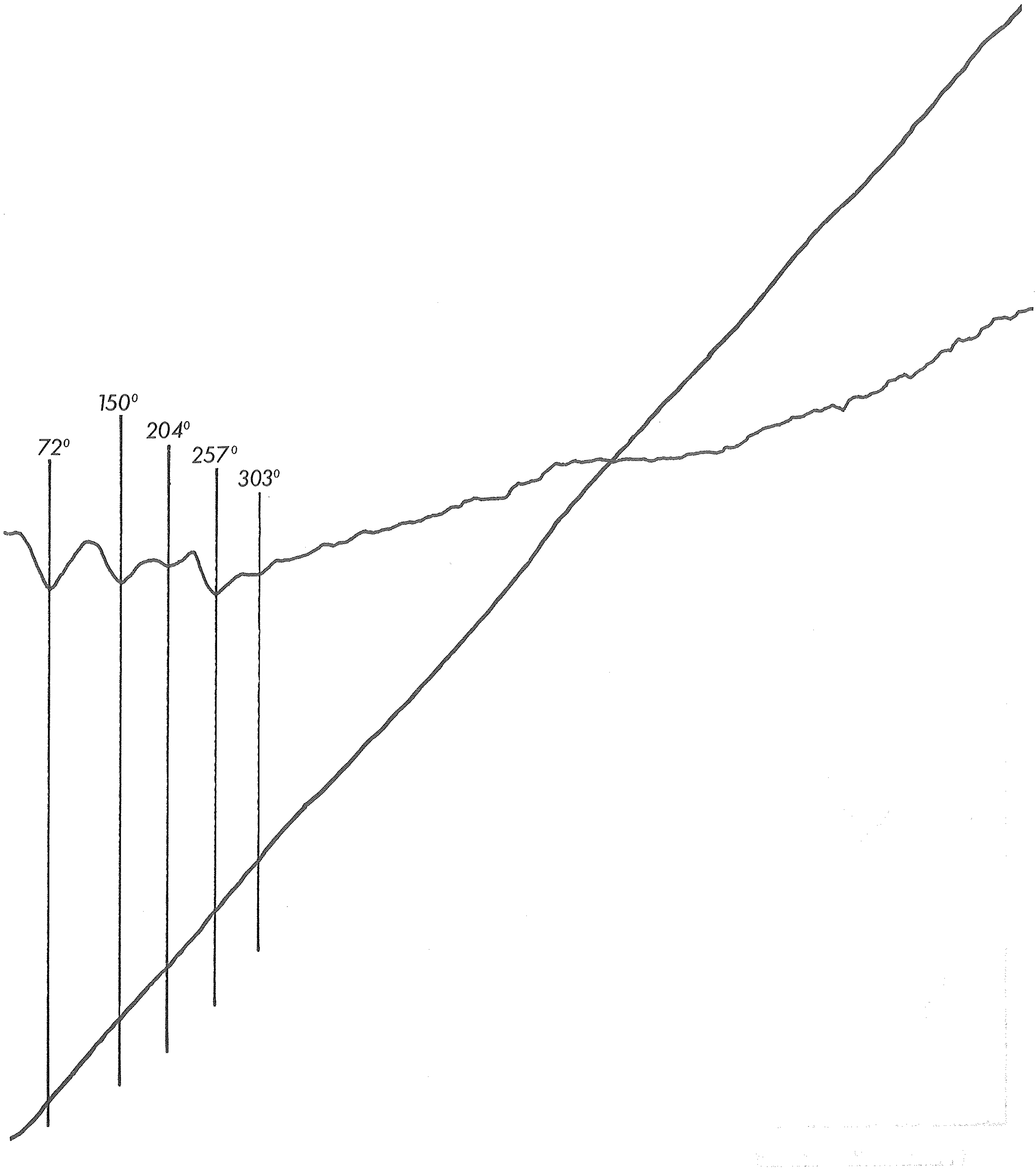
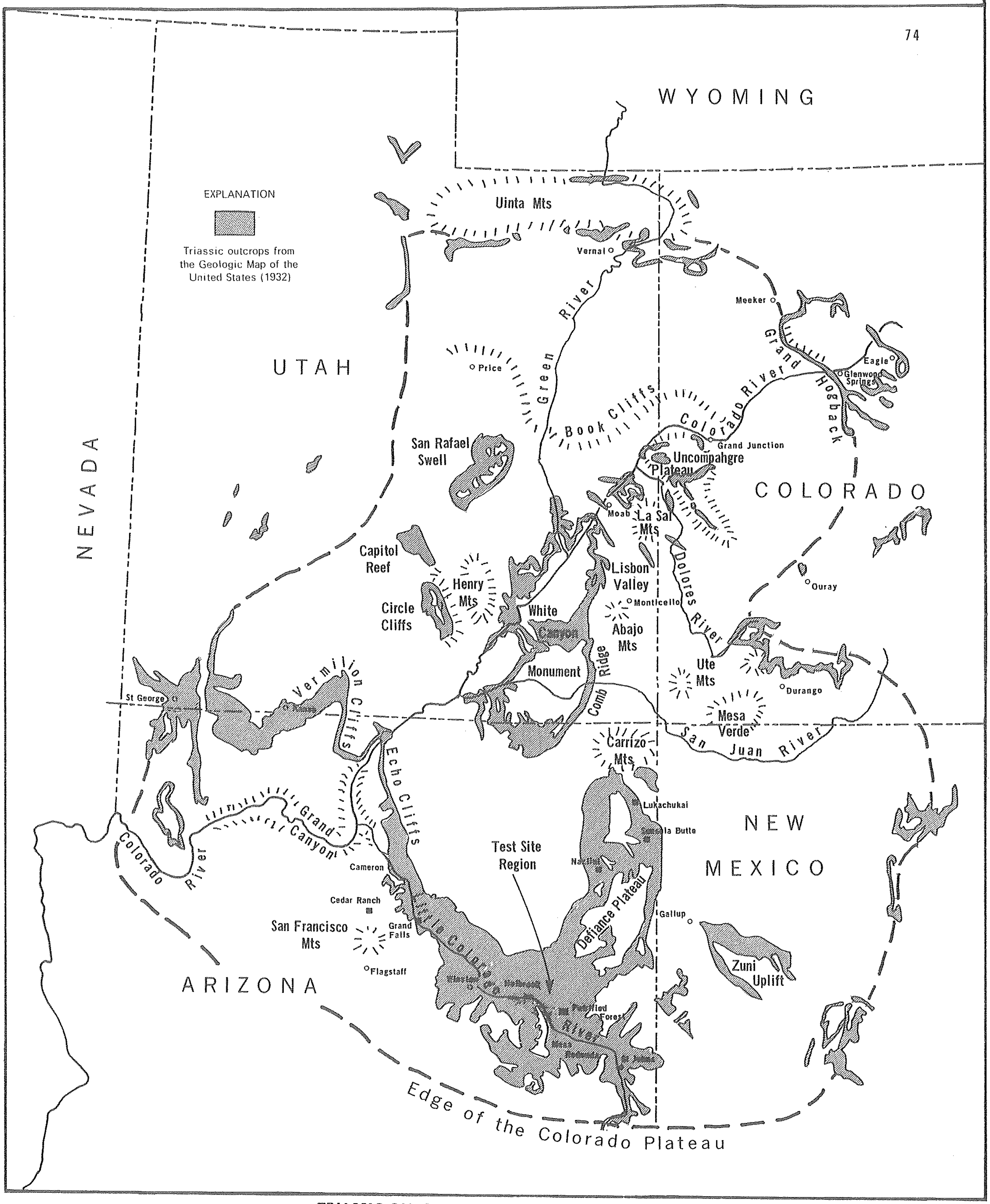


FIG. 6



TRIASSIC OUTCROPS ON THE COLORADO PLATEAU

Plate 1

0 25 50 75 100 MILES